

The corrections requested by the Examiner on page 2 of the rejection have been made.

The allowance of claim 50 and the indication of allowability of claims 11, 16, 52 and 53 is gratefully acknowledge by applicants.

Claims 5-10, 18, 24-32, 34-38, 40-43 and 47 have been rejected under 35 USC 103 over Murai et al.

The Examiner asserts that the C<sub>5</sub> and C<sub>6</sub> alkyl species of the present claims would have been obvious from the C<sub>1</sub>-C<sub>4</sub> progression of the Murai et al patent.

Claims 5-10, 14, 18, 24-32, 34-38, 40-43, 47 and 51 are rejected under 35 UCS 103 as being unpatentable over Matsumura.

Claim 45 has been rejected under 35 USC 103 as being unpatentable over Saeki.

Applicants respectfully request reconsideration of the these rejections by the Examiner in view of the amendments to the claims and the hereinbelow remarks.

The present claims have been amended so as to exclude those compounds wherein R<sub>1</sub> is a C<sub>3</sub>-C<sub>6</sub> alkyl substituted by phenyl.

In regard to these rejections, applicants wish to direct the Examiner's attention to the CCPA entitled In re Mckellin, 188 USPQ 42B. It is believed that the decision is directly on point herein and indicates that the section 103

rejections made by the Examiner are improper.

The interference proceedings in the present application involved specific species which are no longer encompassed by the present claims. In the present application, the interferences were decided on the basis of foreign priority dates. The lost counts of the interference are prior art only under 35 USC 102(g). An award based upon prior invention in foreign country is not prior art under 35 USC 103. A rejection under section 103 "on the theory that an applicant who has lost an interference can never be entitled to claims which are obvious variations of the invention defined in the lost counts" in the case when priority was based on foreign priority was found to be without any foundation in judicial doctrine (see pg. 435 of the decision).

Withdrawal of these rejections and allowance of the present claims is requested.

Claims 45 has been cancelled.

Claims 5-9, 18, 24-32, 34-38, 40-43, 45 and 47-49 have been rejected under 35 USC 112.

The corrections requested by the Examiner have been made. Sulfur should have been "sulfo".

It is believed the claims are in condition for allowance and notice thereof is respectfully requested.

Respectfully submitted,

SPRUNG HORN KRAMER & WOODS

ECR:sm

600 Third Avenue  
New York, New York 10016  
(212) 661-0520

I hereby certify that this correspondence is being deposited with the United States Postal Services as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks, Washington, D.C. 20231 on 3/12/86  
SPRUNG HORN KRAMER & WOODS

By Eugene C. Rzuca  
Eugene C. Rzuca  
Reg. No. 31,900

By Sybil A. Kene

Date 3/12/86

Part 9  
#  
34

testimony is credited, it appears that Piher will prevail. But the question of credibility and the interpretation of the exhibits are matters that must be decided in the first instance by a trial judge. We therefore remand for a new trial of all issues raised by Piher's on sale challenge to the validity of the '285 patent.

#### V.

As already noted, CTS has never marketed its "low cost 450" control commercially. Moreover, there were defects, albeit correctable, in the prototypes which CTS tested. Piher therefore argues that the invention was not useful, that the best mode of carrying it out was not described in the '285 patent specifications, and that the invention was abandoned. The trial judge rejected each of these contentions and we cannot say that his findings are clearly erroneous.

[10] The fact that there was a defect in the prototypes surely does not demonstrate that the *invention* was not useful. Indeed, since the basic features of the invention appear to be embodied in Piher's PT-15 trimmer — as may fairly be inferred from Piher's on sale defense and CTS' original charge that the PT-15 infringes the '285 patent — and since Piher's trimmer is evidently a commercial success, it seems logical to infer that the subject matter of the invention is useful within the meaning of §102.

[11] Similarly, even if the CTS prototypes are not the best possible embodiment of the invention, we find nothing in the record to support the argument that the inventors contemplated a better mode than that disclosed in the specifications. Section 112 merely requires that the patent disclose "the best mode contemplated by the inventor of carrying out his invention."

[12] Finally, the fact that CTS promptly filed its patent application forecloses the contention that the invention was abandoned within the meaning of §102(c). There are, of course, cases in which the character of the commercial exploitation of an invention will be relevant to the issue of abandonment, cf. *Dunlop Holdings, Ltd. v. Ram Golf Corp.*, — F.2d —, No. 74-2024 at 6 (7th Cir., Oct. 20, 1975), but if the application is promptly filed and diligently prosecuted, the decision to postpone commercial development does not constitute abandonment.

#### VI.

The district court refused to award costs to CTS, even though it prevailed on all issues decided by the district court. CTS has therefore filed a cross appeal, relying heavily

on our recent decision in *Popeil Bros., Inc. v. Schick Electric, Inc.*, 516 F.2d 772, 186 USPQ 244 (1975). In view of our remand for a new trial of the on sale defense, we believe that the question of what costs, if any, CTS should recover, can await the conclusion of the proceedings in the trial court.

The judgment of the district court is affirmed in part and reversed in part.

### Court of Customs and Patent Appeals

In re McKellin, Mageli, and D'Angelo

No. 75-539 Decided Jan. 22, 1976

#### PATENTS

##### 1. Patentability — Anticipation — Involving interference (§51.213)

##### Pleading and practice in Patent Office — Rejections (§54.7)

Applicant who lost interference is not entitled to claims corresponding to lost counts' subject matter; determination of priority adverse to applicant constitutes final refusal of claims involved.

##### 2. Court of Customs and Patent Appeals — Issues determined — In general (§28.201)

Although arguments put forth for first time in Court of Customs and Patent Appeals will not ordinarily be considered, court elects to consider new argument that has guidance potential and will not prejudice applicants.

##### 3. Interference — In general (§41.01)

35 U.S.C. 135(a) is procedural rather than substantive.

##### 4. Interference — Practice (§41.60)

##### Patentability — Anticipation — Involving interference (§51.213)

##### Pleading and practice in Patent Office — Rejections (§54.7)

35 U.S.C. 135(a) provides procedure to economize time and work in further prosecution of losing party's application; claims' final refusal may be based on statutory prior art, loss of right to patent, or estoppel; 35 U.S.C. 135(a) is not statutory prior art section, and neither counts nor

their subject matter is statutory prior art under section.

**5. Court of Customs and Patent Appeals — Issues determined — Ex parte patent cases (§28.203)**

**Patentability — Invention — In general (§51.501)**

**Pleading and practice in Patent Office — Rejections (§54.7)**

Court of Customs and Patent Appeals discusses propriety of examiner's positions that were not specifically reversed by Board of Appeals; 35 U.S.C. 103 rejection must be based on evidence, which could include statutory prior art and admissions against interest.

**6. Patentability — Anticipation — Combining references (§51.205)**

**Patentability — Anticipation — Involving interference (§51.213)**

Loss of interference counts alone does not make their subject matter statutory prior art to losing party; lost counts' subject matter that is Section 102 prior art may be used alone or in combination with other references under Section 103.

**7. Interference — Priority (§41.70)**

Award of priority to interference party implies that he made counts' invention prior to filing date of adverse party's parent application.

**8. Court of Customs and Patent Appeals — Issues determined — Ex parte patent cases (§28.203)**

Court of Customs and Patent Appeals reviews Board of Appeals decision on stated ground of rejection and not merely on reasoning supporting decision.

**9. Disclaimer — In general (§32.1)**

**Interference — Conceding priority (§41.15)**

**Interference — Evidence — Failure to take (§41.357)**

**Patentability — Anticipation — Involving interference (§51.213)**

Applicant's refusal to copy claims that he could make, and preventing priority award based on evidence, results in concession of priority, or "disclaimer," so that claims' subject matter is Section 102(g) prior invention of another in this country and prior art to applicant under Section 103 where concession of priority does not depend on when or where subject matter was invented; however, facts will not be inferred that contradict facts of record forming basis of

priority award; consequences ensuing from priority award based on evidence of record significantly differ from inferences made in absence of evidence.

**10. Patentability — Anticipation — In general (§51.201)**

**Patentability — Invention — In general (§51.501)**

Prior invention in this country is prior art under Sections 102(g) and 103, but prior invention in foreign country is not Section 103 prior art.

**11. Patentability — Anticipation — Involving interference (§51.213)**

**Patentability — Anticipation — Patents — Foreign (§51.2215)**

**Patentability — Anticipation — Patents — On copending applications (§51.2219)**

Lost counts' subject matter that was neither known nor used by others in this country before applicant's effective filing date, nor patented or published before effective filing date, is not Section 102(a) prior art; foreign application that was not filed more than one year before applicant's effective filing date is not Section 102(b) prior art; effective date of patent's disclosure vis-a-vis U.S. application is its U.S. filing date; disclosure of patent whose U.S. filing date is subsequent to applicant's effective date is not Section 102(e) prior art.

**12. Interference — Conceding priority (§41.15)**

**Patentability — Anticipation — Involving interference (§51.213)**

Conceding priority of counts' subject matter is admission that subject matter is Section 102(g) prior invention of another in this country and Section 103 prior art, absent evidence of where invention was made; applicant's concession that counts' subject matter is prior art renders it inappropriate to question use of subject matter as if it were prior art with respect to applicant's claims.

**13. Interference — Priority (§41.70)**

**Patentability — Anticipation — In general (§51.201)**

**Patentability — Anticipation — Patents — Foreign (§51.2215)**

**Patentability — Invention — In general (§51.501)**

Giving invention apparently made outside U.S. same effective date as reference as if it had been made in this country is contrary to statutory limitations.

#### 14. Patentability — Anticipation — Involving interference (§51.213)

##### Patentability — Invention — In general (§51.501)

##### Pleading and practice in Patent Office — Rejections (§54.7)

No judicial doctrine supports 35 U.S.C. 103 rejection on theory that applicant who lost interference is never entitled to claims that are obvious variation of invention defined in lost counts.

##### Particular patents — Vulcanizing

McKellin, Mageli, and D'Angelo, New Vulcanizable and Vulcanized Compositions Containing Polyperoxide, rejection of claims 6, 7, 10-18, and 21-25 reversed.

##### Appeal from Patent and Trademark Office Board of Appeals:

Application for patent of Wilbur H. McKellin, Orville L. Mageli, and Antonio Joseph D'Angelo, Serial No. 866,420, filed Sept. 30, 1969, continuation in part of Serial No. 285,857, filed June 6, 1963. From decision rejecting claims 6, 7, 10-18, and 21-25, applicants appeal. Reversed; Markey, Chief Judge, and Rich, Judge, concurring with opinions; Miller, Judge, dissenting with opinion.

Carl A. Hechmer, Jr., Philadelphia, Pa. (Howson & Howson, William E. Hedges, and Charles H. Howson, Jr., all of Philadelphia, Pa., of counsel) for appellants.

Joseph F. Nakamura (Jack E. Armore, of counsel) for Commissioner of Patents and Trademarks.

Before Markey, Chief Judge, and Rich, Baldwin, Lane, and Miller, Associate Judges.

Lane, Judge.

This is an appeal from the decision of the Patent and Trademark Office Board of Appeals affirming the examiner's rejection of claims 6, 7, 10-18, and 21-25, all of the claims in application serial No. 866,420, filed September 30, 1969, for "New Vulcanizable and Vulcanized Compositions Containing Polyperoxide." We reverse.

##### Background

Appellants' application returned to ex parte prosecution after appellants lost an interference with United States Letters Patent 3,409,600 to P.R.A. Maltha and S.B. Tijssen. None of the claims on appeal were counts. The sole basis of rejection is under

35 USC 103, the rejected claims being held obvious in view of the subject matter of the counts of the interference. The sole issue on appeal is whether claims may be rejected under 35 USC 103 on the ground that a losing party to an interference is not entitled to claims which are asserted to be obvious variations of the invention defined in the counts, when section 102(g) and interference estoppel are not applicable.

##### The Interference

Appellants' parent application (serial No. 285,857) was filed in the United States Patent and Trademark Office on June 6, 1963. Subsequently, on October 10, 1963, Maltha et al. (hereinafter Maltha) filed a patent application, a division of which issued as United States Letters Patent 3,409,600 on November 5, 1968. The Maltha patent claims a process and compositions within the scope of serial No. 285,857, in the sense that an act which infringes the claims of the Maltha patent would infringe the claims of serial No. 285,857. Appellants thereafter filed a continuation-in-part application, serial No. 866,420, which is involved in this appeal. This application contained claims copied from the Maltha patent and included a request for an interference. All of the claims of the Maltha patent were copied. The class of peroxy compounds disclosed and for which protection was sought in serial No. 866,420 was broader (i.e., included more species) than the class of peroxy compounds disclosed and claimed in the Maltha patent.

Thereafter patent interference No. 97,329 was declared between Maltha and the appellants herein. Maltha was accorded the benefit of his Netherlands application No. 284,315, filed October 12, 1962. Based *only* on their foreign priority date Maltha became the senior party and prevailed in the interference, receiving an award of priority.

During the course of the interference appellants moved to add additional counts to include species disclosed in the patent which were within the scope of the interference counts and also to add counts to species which were not disclosed in the patent but were within the scope of the interference counts. These motions were refused entry by the interference examiner.

Following the adverse award of priority, appellants' application returned to ex parte prosecution with claims directed to the species just described, as well as claims to other species neither disclosed in the patent nor within the scope of the interference counts. All of these claims differ from the interference counts in one or more material respects. Claims 6 and 13, which are

claims being held subject matter of the patent. The sole issue on which the claims may be rejected is the ground that a loss of novelty is not entitled to be obvious under the definition in the patent 102(g) and interference is not applicable.

Interference (serial No. 97,329) United States Patent Office on June 6, 1963. On October 10, 1963, Maltha et al. filed a patent application of which issued as Patent 3,409,600 on October 14, 1968. Maltha patent is in opposition within 35 USC 102(g), in the sense that the claims of the Maltha patent thereafter filed in application, serial No. 97,329, involved in this interference contained claims patent and included interference. All of the claims of the patent were copied. The compounds disclosed in the patent were sought in the prior art (i.e., including the class of peroxy compounds claimed in the

Interference No. 97,329 Maltha and the patent was accorded the priority date of the application No. 97,329. Based on the priority date Maltha patent prevailed in the award of priority. In the interference additional counts were added in the patent scope of the invention to add counts to the scope of the invention as disclosed in the patent scope of the invention. The motions were refused by the examiner.

Award of priority, turned to ex parte directed to the patent as well as claims to be added in the patent scope of the invention. The interference is later from the interference more material 102(g), which are

representative, are reproduced in the appendix to this opinion together with the interference counts.

#### *The Examiner's Rejection*

The examiner rejected the claims under 35 USC 103 as unpatentable in view of the counts of the interference, or in view of the Maltha patent. The examiner concluded that the instant claims would be obvious to one of ordinary skill in the art from the counts of the interference. Appellants have made no attempt to prove any new or unexpected results.

#### *The Board's Decision*

The board interpreted the reasoning of the examiner to mean that the subject matter sought to be patented was barred to appellants by reason of the adverse award of priority as to the counts of the interference.

The board agreed with appellants that if an interference involves two applications, a party who fails to make a motion to add all common subject matter may be estopped from later obtaining claims to such subject matter, but that if (as here) an interference involves a patent, there is no estoppel.

The board considered appellants' argument that since appellants' application had an earlier effective United States filing date than the Maltha patent, from which the claims which corresponded to the interference counts were copied, the counts could not be considered "prior art" under 35 USC 102(g).

The board answered by saying:

We specifically indicate that the rejection is not that the claims are unpatentable over [sic, under] 35 USC 102(g) coupled with 35 USC 119. Rather, the rejection is on the basis that appellant [sic] has lost the interference and, in view of the adverse decision on priority, is not entitled to claims which correspond to or are obvious variations of the invention as defined in the counts of the interference. This is the line of reasoning advanced by the third member of the Board in the Hilmer case [In re Hilmer, 57 CCPA 985, 424 F.2d 1108, 165 USPQ 255 (1970), hereinafter Hilmer (II)], \* \* \* where, while concurring in the result, he stated, "I see no reason to go beyond the concession of priority filed by Hilmer et al. —".

<sup>1</sup> This concurring opinion reads in toto:

Behrens, Examiner-in-Chief, Concurring. As to the affirmance of the rejection now considered, that of claims 10 and 16, I concur in the result. On the record I see no reason to go

Since his view was not determinative of the appeal, the court limited itself to only the correctness of the reasoning of the majority.<sup>2</sup>

The board noted that appellants had not alleged that their invention produced any new or unexpected result over that produced by the invention of Maltha represented by the lost counts, and held that appellants' claims "either fall within the scope of the invention defined by the counts or are obvious variations thereof." Appellants contended there is no evidence of record that the invention defined in the counts was made in this country by another before appellants' invention. The board responded:

If we were to follow appellants' reasoning to its logical conclusion, we would condone a situation in which, by presenting claims, drawn sequentially, to compounds containing carbon chains from 1 to 20 carbon atoms, or to obvious variations of the compounds, the losing party in an interference with a patent, (which relied on a foreign priority date), could be granted a patent which would cover all aspects of the invention defined in the counts of the interference as to which he has been adjudicated not to be the first inventor. Obviously, this would make a mockery of the interference practice.

#### *The Solicitor's Position*

The solicitor, representing the Commissioner of Patents and Trademarks, has taken the position that the counts which appellants lost in the interference are "prior art" to appellants. It is the solicitor's position that having lost the counts in an interference, the subject matter of the lost counts is statutory prior art to the losing party under 35 USC 135(a). The solicitor observes that section 135 provides that the

beyond the concession of priority filed by Hilmer et al. in Interference No. 90,218 which applied to the count corresponding to Hilmer et al. claim 15. The art, as Wagner et al. show, recognized the equivalence of the toluyl (of claim 15) and cycloaliphatic radicals (of claims 10 and 16) in compounds very closely related as to structure and intended use. Appellants establish no earlier date of invention for the cycloalkyl compounds than for their toluyl analogues on which they have conceded priority. In re Gregg, 44 CCPA 904, 1957 C.D. 284, 720 O.G. 227, 244 F.2d 316, 113 USPQ 526 [(1957)].

<sup>2</sup> See Hilmer (II), 57 CCPA at 987, 424 F.2d at 1110, 165 USPQ at 257.

losing party is not entitled to claim the subject matter of the lost counts. Therefore, the solicitor reasons, the subject matter of the lost counts is statutory prior art to the losing party in the same anticipatory sense as the statutory prior art described in 35 USC 102, and also within the meaning of the term "prior art" in 35 USC 103.

### Opinion

#### Section 135

[1] An applicant who has lost an interference is not entitled to claims which correspond to the subject matter of the counts of the interference. A determination of priority of invention adverse to an applicant constitutes the *final refusal* by the Patent and Trademark Office of the claims involved. 35 USC 135. Neither party contends that the claims on appeal "correspond" to the counts in the interference.

[2] We turn now to the statutory construction of 35 USC 135(a) which the solicitor here presents to this court as a novel proposition of law and for the first time in the prosecution of appellants' application. As the solicitor is well aware, this court will not ordinarily consider arguments put forward for the first time in this court. In re Allen, 51 CCPA 809, 815, 324 F.2d 993, 998, 139 USPQ 492, 496 (1963). Nevertheless, we have elected to consider this argument in the present case because of its guidance potential and because our view thereof cannot prejudice appellants.

[3] The solicitor suggests that the provision in 35 USC 135(a) that an adverse award of priority in an interference "shall constitute the final refusal by the Patent and Trademark Office of the claims involved" means that the subject matter of the counts is *statutory prior art* to the losing party in the same anticipatory sense as the statutory prior art described in section 102 and also within the meaning of the term "prior art" in section 103. Nothing in the legislative history of 35 USC 135(a) supports the solicitor's position. The Reviser's Note, 35 USCA 135, explains:

The first paragraph states the existing corresponding statute [R.S. 4904] with a few changes in language. An explicit statement that the Office decision on priority constitutes a final refusal by the Office of the claims involved, is added.

The Commentary on the New Patent Act, by P.J. Federico, 35 USCA at 37-38, confirms this, and states further, "The sections just mentioned, 131 to 135, deal with *procedure* in the Patent Office, of which only a

small part is reflected in the statute." (Emphasis added.) A reading of section 135(a) confirms that all its provisions relate to procedure rather than substance.

[4] In support of his statutory construction, the solicitor reasons that because the claims corresponding to the counts are unpatentable to the losing party, the subject matter of the counts *must be statutory prior art* to the losing party. The weakness of this reasoning is apparent. The purpose of section 135(a) was, in part, to provide a new procedure to economize time and work in the further prosecution of the losing party's application. The final refusal of claims by the Patent and Trademark Office may be based, inter alia, on statutory prior art or loss of right to a patent or an estoppel. The inference that the counts (i.e., the subject matter of the counts) must be statutory prior art to the losing party merely because section 135(a), as a matter of Patent and Trademark Office procedure, provides for automatic "final refusal" of claims corresponding to the counts by virtue of the adverse award of priority, is unwarranted. We specifically reject the novel proposition that 35 USC 135(a) is a statutory prior art section, and hold that neither the counts nor the subject matter of the counts is statutory prior art by virtue of this section of the statute.

#### Section 103

[5] The examiner rejected the claims under 35 USC 103 as unpatentable in view of the counts of the interference, or in view of the Maltha patent. Since the examiner's position was not specifically reversed by the board (37 CFR 1.196(a)), we shall discuss the propriety of these positions. In re Halliwell, 53 CCPA 1112, 358 F.2d 1008, 149 USPQ 511 (1966). It was the examiner's position that the subject matter of the appealed claims would have been obvious at the time appellant's invention was made to a person having ordinary skill in the art in view of the counts of the interference, i.e., the subject matter of the counts, see Hilmer (II), 57 CCPA at 987, 424 F.2d at 1110, 165 USPQ at 256-57, or in view of the disclosure of the Maltha patent. Since a section 103 rejection must be based on *evidence* (statutory prior art, admissions against interest, etc.), we shall first consider the status of the subject matter of the counts lost in the interference, and the disclosure of the Maltha patent, as statutory prior art with respect to the appealed claims.

[6] The subject matter of counts lost in an interference is not, for that reason alone, statutory prior art to the losing party. We



in the statute." Reading of section 102 provisions relate to substance.

statutory constructions that because the counts are un- party, the subject matter of the statutory prior art is the weakness of this

The purpose of section 102 is to provide a new time and work in of the losing party's refusal of claims by Patent Office may be statutory prior art or an estoppel. The counts (i.e., the subject matter) must be statutory prior art merely because matter of Patent and procedure, provides for of claims counts by virtue of the right is unwarranted. the novel proposition of a statutory prior art neither the counts nor the counts is statutory prior art this section of the

rejected the claims un- preventable in view of interference, or in view of since the examiner's decision reversed by the (a)), we shall discuss these positions. In re 1012, 358 F.2d 1008, 1009. It was the ex- the subject matter of could have been ob- dent's invention was ordinary skill in the of the interference, of the counts, see at 987, 421 F.2d at 1007, or in view of the patent. Since a sec- be based on evidence decisions against in- consider the status of the counts lost in the disclosure of the statutory prior art with d claims.

of counts lost in for that reason alone, the losing party. We

must therefore consider whether the subject matter of the counts lost in the interference falls within a prior art paragraph of section 102. If it is found that the subject matter of the lost counts is prior art under section 102, it may be used alone or in combination with other references under section 103. In re Harry, 51 CCPA 1541, 333 F.2d 920, 142 USPQ 164 (1964).

[7] We must first determine when and where the subject matter of the lost counts was invented in order to ascertain the significance of the lost counts with respect to section 102. Appellants' parent application was filed June 6, 1963. Appellants were restricted to that date by the Board of Patent Interferences. The decision of the board awarding priority to Maltha necessarily implies that Maltha made the invention of the counts prior to June 6, 1963. We conclude that the date of the invention of the subject matter of the lost counts must be prior to June 6, 1963. The place where Maltha made the invention of the counts appears from the evidence to have been outside the United States, since Maltha was awarded priority on the basis of his Netherlands filing date.

[8] The board's opinion in the present case indicates reliance on the line of reasoning advanced in the concurring opinion of the Board of Appeals in Hilmer (II), set forth in footnote 1, supra. The board stated that because the reasoning advanced in the concurring opinion in Hilmer (II) was not determinative of that appeal, this court reviewed only the correctness of the reasoning of the majority of the board. Our analysis should not be confined to the board's reasoning alone since we review the decision of the board on the stated ground of rejection and not merely its reasoning in support of that decision. *Hunt v. Treppschuh*, 523 F.2d 1386, 1388, 187 USPQ 426, 428 (CCPA 1975).

[9] We have recently reviewed the consequences of a concession of priority with respect to the count of an interference. See *In re Ogilvie*, 517 F.2d 1382, 186 USPQ 227 (CCPA 1975). Therein we held that an applicant's refusal to copy claims which he could make resulted in a concession of priority (termed a "disclaimer" in 37 CFR 1.203(b)) that the subject matter of those claims is the prior invention of another in this country under section 102(g) and thus prior art to that applicant under section 103. *Id.* at 1390-91, 186 USPQ at 234-35. That is the legal consequence of a concession of priority which does not depend on when or where the subject matter of the claims not copied was invented. *Id.* at 1391 n.3, 186 USPQ at 235 n.3. Such consequences are imposed when an applicant, by refusing to copy claims,

prevents an award of priority based on evidence of record. However, when, as in the present case, priority is awarded on the basis of evidence of record, the law will not infer facts which are contradicted by facts of record. The evidence of record in the present case consists of the factual allegations in the motion of Maltha to be accorded the benefit of his Netherlands filing date, and to shift the burden of proof. This motion, supported by the necessary documents (37 CFR 1.224), granted by the primary examiner and the Board of Patent Interferences, is evidence, accepted by the Patent and Trademark Office, that Maltha made the invention of the counts outside the United States. Because of the significantly different consequences which may ensue from an award of priority based on evidence of record, as opposed to inferences made in the absence of evidence, we find the board's reliance on a theory of concession of priority inapposite in the present case.

[10] The board's opinion concludes with the statement set forth above, critical of the significantly different consequences which may ensue from an award of priority based on prior invention in this country, which is prior art under section 102(g) and section 103, as opposed to an award based on prior invention in a foreign country, which is not prior art under section 103. As we said in *Hilmer (II)*, if the law as it has been written by Congress creates anomalous situations, then it is for Congress to decide whether to change the law. See *Hilmer (II)*, 57 CCPA at 991 n.6, 424 F.2d at 1113 n.6, 165 USPQ at 259 n.6. We are aware that Congress is currently considering legislation to amend the patent statute. It may well wish to consider whether it would be desirable to amend Title 35 so that a foreign priority application is for all purposes given the status of an application filed in the United States on the day it was first filed in a foreign country, a matter on which we express no opinion.

[11] We have considered whether the subject matter of the counts lost in the interference falls within any "prior art" paragraph of section 102 other than section 102(g), and find that it does not. The subject matter of the lost counts is not prior art under section 102(a) because it was neither known nor used by others in this country before appellants' effective filing date. Further, there is no evidence that the invention disclosed in Maltha's Netherlands application No. 284,315, filed October 12, 1962, was either patented or published before appellants' effective filing date. With respect to section 102(b), Maltha's Netherlands application No. 284,315 was

not even filed more than one year before appellants' effective filing date, June 6, 1963.

With respect to section 102(c), the effective date of the disclosure of the Maltha patent vis-a-vis an application filed in the United States is the United States filing date of Maltha. In re Hilmer, 53 CCPA 1288, 1318-20, 359 F.2d 859, 882-83, 149 USPQ 480, 499-500 (1966), hereinafter Hilmer (I). The United States filing date of the Maltha patent is subsequent to appellants' effective date. Therefore the disclosure of the Maltha patent cannot be prior art under section 102(c).

There is no other statutory basis for finding that either the subject matter of the lost counts or the disclosure of the Maltha patent is prior art, in the sense of 35 USC 103, to appellants.

#### *Obvious Variations of the Lost Interference Counts*

The board noted that appellants have neither introduced any evidence of unobvious results, nor have they even argued the question of obviousness. We assume, without deciding, that the claims on appeal are directed to obvious variations of the invention of the counts. The question remains whether there is any judicial doctrine which will support a rejection under 35 USC 103 of such claims.

The solicitor has directed our attention to several cases which involve rejection of claims of a party losing an interference, using the counts of the interference as prior art. None of these cases involved an interference decided on the basis of the winning party's right to the benefit of an earlier foreign filing date under 35 USC 119, or corresponding earlier statutory provisions. We briefly review each of the cases cited by the solicitor.

[12] In In re Cole, 23 CCPA 1057, 82 F.2d 405, 29 USPQ 137 (1936) this court held that when an applicant has conceded that he is not entitled to a patent for the subject matter embraced in the interference counts, claims thereafter presented must be "inventively different" from the interference counts. The court in Cole relied on In re Williams, 20 CCPA 738, 62 F.2d 86, 16 USPQ 66 (1932); In re Dodge, 22 CCPA 870, 74 F.2d 756, 24 USPQ 219 (1935); and In re Sola, 22 CCPA 1313, 77 F.2d 627, 25 USPQ 433 (1935), which are also relied on by the solicitor here. In Williams, after the declaration of the interference, the appellants filed a disclaimer and canceled their claims which corresponded to the interference counts, which was held to have been an admission that they were not entitled

to the counts and that they were also not entitled to any subject matter failing to "define invention" thereafter. In Dodge the subject matter of the interference counts was conceded by the appellant to be in the prior art and hence unpatentable to anyone. In Sola the appellant conceded priority of invention. As we discussed in Ogiue, supra, absent evidence of where the invention was made, conceding priority of the subject matter of counts is an admission that such subject matter is the prior invention of another in this country under section 102(g) and thus prior art under section 103. Further, when an applicant, as in Dodge, concedes the subject matter of the interference counts is in the prior art, it is inappropriate to question the propriety of using such subject matter as if it were prior art with respect to claims presented by that applicant. See In re Hell-sund, 59 CCPA 1382, 1387, 474 F.2d 1307, 1311, 177 USPQ 170, 173 (1973); In re Nomiya, 509 F.2d 566, 570-71, 184 USPQ 607, 611-12 (CCPA 1975).

In In re Karplus, 25 CCPA 1192, 97 F.2d 100, 37 USPQ 800 (1938) this court relied upon the holding in Cole to reject claims as not "patentably distinct" over the count of an interference. The Board of Appeals' opinion had noted that the Karplus application had been involved in several interferences, each of which was terminated by dissolution rather than by any award of priority. From the Karplus Petition for Rehearing it appears that the subject matter of the count had, during the interference, been held to be in the prior art and therefore unpatentable to anyone. Karplus was estopped by the interference judgment to assert that the subject matter of the count was not in the prior art.

The appeal in In re Fenn, 50 CCPA 1163, 315 F.2d 949, 137 USPQ 367 (1963) followed an interference which had been dissolved after appellant filed an abandonment of the contest, 37 CFR 1.262(b). See Schnick v. Fenn, 47 CCPA 1174, 1178 n.3, 277 F.2d 935, 938 n.2, 125 USPQ 567, 570 n.2 (1960). Under 37 CFR 1.262(b), this court held that the appellant stood in the same position as he would have, had there been an award of priority adverse to him with respect to the interference count. The issue presented was whether the appealed claim distinguished patentably from the interference count. 50 CCPA at 1165, 315 F.2d at 950-51, 137 USPQ at 368. Both parties in Schnick v. Fenn, supra, had worked in this country. Therefore, with respect to Fenn, the subject matter of the count may be considered to have been the prior invention of another made in this country.

were also not entitled to "define" the subject matter as to anyone. In *Sola*, supra, absent mention was made of the subject matter of that such subject matter of another in the prior art. Further, when an invention is made, the subject matter is in the prior art. To question the subject matter as if it were not in the prior art. See *In re Hellmuth*, 377 F.2d 1307, 1373 (1973); *In re* 1970-71, 184 USPQ 1192, 97 F.2d 1192. This court relied on the fact that the board rejected claims as to the count of the Appeals' opinion plus application of the interference, and by dissolution of the priority. From the rehearing it was held that the number of the count had been held to be unpatentable to the board by the invention that the subject matter was not in the prior art.

In 50 CCPA 1163, 1377 (1963) following the dissolution of the board. See *Schnick v. ...*, 377 F.2d 1307, 1373 n.3, 277 F.2d 1307, 570 n.2 (1960). The court held that the same position as had been an award of the subject matter with respect to the invention presented was the same distinguished interference count. 50 CCPA at 950-51, 1377. In this country, the subject matter is to be considered to be the subject matter of another

[13] Finally, the solicitor directs our attention to a portion of the principal opinion of the court in *In re Bass*, 59 CCPA 1342, 1359, 474 F.2d 1276, 1289-90, 177 USPQ 178, 188 (1973). In the page referred to by the solicitor it is first stated that prior invention under section 102(g) is prior art. The facts of the present case do not comply with the "in this country" provision of section 102(g) and it is therefore inapplicable. The page referred to by the solicitor then distinguishes the facts of *Bass* from the facts of *Hilmer* (I). The facts of the present case are analogous to the facts of the *Hilmer* cases in that here, as there, we are asked to give an invention apparently made outside the United States the same effective date as a reference as it would have had, had it been made in this country. This is contrary to what we have found to be the express desire of Congress and statutory limitations to events which take place in the United States. We find nothing in the page referred to by the solicitor which is relevant to the present case.

[14] The board based the rejection under 35 USC 103 on the theory that an applicant who has lost an interference can never be entitled to claims which are obvious variations of the invention defined in the lost counts. We find no judicial doctrine which supports this rejection under 35 USC 103.

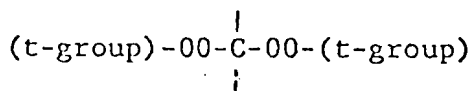
#### Conclusion

The decision of the board affirming the rejection of claims 6, 7, 10-18, and 21-25 is reversed.

#### Appendix

##### The Subject Matter

The subject matter of the claims is a vulcanizable composition comprising a mixture of a polymeric compound capable of being cross-linked to form a thermoset material, and a cross-linking agent in an amount sufficient to effect the desired degree of cross-linking. The cross-linking agent is a polyperoxy compound having at least two peroxy groups (-OO-). One oxygen atom of each of two of these peroxy groups is linked to a common carbon atom. The other oxygen atom of each of these two peroxy groups is linked to a tertiary carbon or silicon atom of an organo group. A simplified drawing of one pair of these peroxy groups may be illustrated as:



The common carbon atom is part of an aliphatic group which has at least two carbon atoms and includes at least one carboxy group. This carboxy group may be present as an acid (-COOH), an ester (-COOR), an

acid anhydride ( $\begin{array}{c} \text{O} \quad \text{O} \\ \parallel \quad \parallel \\ -\text{C}-\text{O}-\text{C}- \end{array}$ ), or a metal salt (-COOM). The cross-linked composition which is the product of heat-curing this vulcanizable composition is also claimed. Claims 6 and 13 are representative:

6. A vulcanizable composition comprising an intimate mixture of a polymeric compound capable of being cross-linked to form a thermoset material, and cross-linking agent in an amount sufficient to afford about the desired degree of cross-linking, which agent is a polyperoxy compound having at least two peroxy groups, one of the oxygens of each of two of said peroxy groups being linked to a common carbon atom and each of the other oxygens being linked to a tertiary atom of an organo member which tertiary atom is selected from the class consisting of carbon and silicon and said common carbon atom being part of an aliphatic group selected from those having 2-3 or 5-6 carbons plus at least one carboxy group and those having 4 carbons plus at least one carboxy group selected from

$\begin{array}{c} \text{O} \quad \text{O} \\ \parallel \quad \parallel \\ -\text{C}-\text{O}-\text{C}- \end{array}$ , where M is a metal ion.

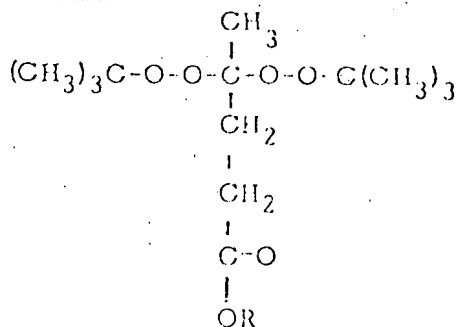
13. A cross-linked composition comprising the product of heat curing a composition comprising an intimate mixture of a polymeric compound selected from the class consisting of solid polyolefins and elastomers and a cross-linking agent in an amount sufficient to convert said polymeric compound to a cross-linking thermoset material, which agent is a polyperoxy compound having at least two peroxy groups (one pair), one of the oxygens in each of said peroxy groups of one pair being linked to a common carbon atom and the other oxygen being linked to a tertiary atom, selected from the class consisting of carbon and silicon, of an organo group selected from the class consisting of aliphatic hydrocarbon, cycloaliphatic hydrocarbon, aralkyl hydrocarbon and silyl having not more than a total of 24 carbon and silicon atoms; said common carbon atom being part of an aliphatic group selected from those (a) having 2-3 or 5-6 carbon atoms, having only saturated linkages between said carbons, plus at least one acid group selected from the class consisting of -COOH; -COOR; -COOM, and

$\begin{array}{c} \text{O} \quad \text{O} \\ \parallel \quad \parallel \\ -\text{C}-\text{O}-\text{C}- \end{array}$ , and (b) having 4 carbon atoms with only saturated linkages between said carbons plus at least one acid group selected from  $-\text{COOH}$ ,

$-\text{COOM}$  and  $\begin{array}{c} \text{O} \quad \text{O} \\ \parallel \quad \parallel \\ -\text{C}-\text{O}-\text{C}- \end{array}$ , where R is a straight chain aliphatic hydrocarbon group having 1-18 carbon atoms and where M is a metal ion.

#### The Interference Counts

1. In a process for cross-linking a copolymer of ethylene and a monomer containing a  $\text{CH}_2=\text{CH}-$  group with the aid of a peroxidic initiator, the improvement comprising employing as an initiator a compound having the general formula

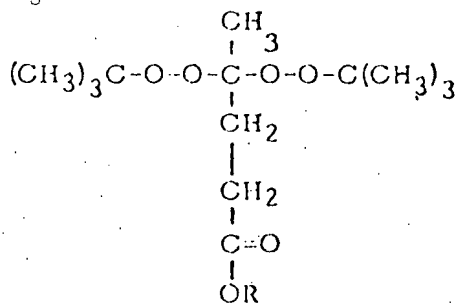


in which R is a hydrocarbon group having not more than 20 carbon atoms.

12. A process as defined in count 1, in which the cross linking is carried out in the presence of at least one added auxiliary agent.

3. An article comprising a cross-linked copolymer of ethylene and a monomer containing a  $\text{CH}_2=\text{CH}-$  group obtained by the process defined in count 1.

4. A new composition of matter comprising a copolymer of ethylene and a monomer containing a  $\text{CH}_2=\text{CH}-$  group, and an organic compound having the general formula



in which R is a hydrocarbon group having not more than 20 carbon atoms.

5. A new composition of matter as defined in count 4 containing in addition at least one added auxiliary agent.

Markey, Chief Judge, concurring.

I join the well-written opinion of the majority. Exercising that which should be rarely exercised, the judicial privilege of additional comment, I append these few remarks.

The board's basis for affirming the rejection was not clearly stated. The board's opinion uses language from 35 USC 103 and fails to indicate reversal of the examiner's reliance thereon. Though the board said it was not employing 35 USC 102(g) coupled with 35 USC 119, it had to have done just that, as indicated below, before it could label the appealed claims "obvious variants."

It seems clear that the limiting phrase "in this country" of 35 USC 102(g) served the basic interests of the United States and the purpose of our patent system i.e., to encourage disclosure here. In earlier, more isolated times, the existence of prior inventions elsewhere in the world, which were not patented or described in a printed publication, was of little or no moment within our borders. Treaty provisions designed to achieve reciprocity, as reflected in 35 USC 119, were not intended to create a new source of "prior art." Whether accelerated communication has so "shrunk" the world as to require that Maltha's invention, on which a patent application had been merely filed in a foreign land, must from its foreign filing date constitute an obstacle to patent-resulting disclosures by our citizens is, I fully agree, a matter for the Congress. The statute, as it now stands, is to the contrary.

Whether 35 USC 135(a) be procedural or substantive, it provides that an adverse interference decision is a final refusal only of "the claims involved." We are not at liberty, it seems to me, to add "or obvious variants thereof" to that phrase in the statute when, as here, the adverse decision rests only on an earlier foreign filing. Hence the very wording of the statute impels the court's rejection of the solicitor's argument built on 35 USC 135(a).

The desire of courts to avoid untoward results has often led to a desirable growth in the life of the law. That desire can also lead to such confusion in the law as to render society almost rudderless in the area involved. Though the law of patents seems occasionally a particular victim, the phenomenon occurs in all types of cases.

hydrocarbon group having carbon atoms.

position of matter as containing in addition auxiliary agent.

lge. concurring.

opinion of the majority which should be rare-  
 lial privilege of ad-  
 I append these few

for affirming the rejection. The board's opinion 35 USC 103 and of the examiner's through the board said it 35 USC 102(g) coupled had to have done just slow, before it could claims "obvious

the limiting phrase "in 35 USC 102(g) served the United States and the patent system i.e., to encourage. In earlier, more existence of prior inventions world, which were not in a printed publication moment within our visions designed to be reflected in 35 USC 102(g) to create a new "Whether accelerated or "brunk" the world had been merely from its foreign obstacle to patent-our citizens is, I full of the Congress. The is to the contrary.

35(a) be procedural or that an adverse in- final refusal only of We are not at liberty, for obvious variants in the statute when, vision rests only on an Hence the very word-ers the court's rejection argument built on 35

to avoid untoward a desirable growth in desire can also lead the law as to render less in the area in- of patents seems oc-ular victim, the all types of cases.

Whether it arises as an element of social jurisprudence or in an effort to effectuate a perceived public policy, the dichotomy between the so-called activists, who would reach for the "proper" result, and the so-called literalists, who would rigidly "follow" the law as written, has probably existed since courts began. An almost infinite shading exists between instances at opposite ends of the spectrum. In seeking a "just" result, the written law may in some cases be only slightly tilted, while in others it may be bent and twisted into unrecognizable and grotesque forms. On the other hand, literal application of statutory law may produce results extending from those only slightly spaced from equity to those so remote from our traditional concepts of justice as to cry out for change in the written law. Similarly, we who are charged with the duty of deciding and of explaining our decisions may, from case to case, often and rightly find ourselves at various wavelengths in the activism-literalism spectrum. Happily, the constant activist, for whom the result controls, and the total literalist, for whom the result is irrelevant, are rare if not extinct.

Though now encrusted with varying and variable judicial interpretations, the patent law is statutory. Our representative form of government requires that the enactments of its Congress must always be, at the very least, the starting point. There being no common law of patents, we should take care to fill the Holmesian interstices of the statute with judge-made law only under the gravest and most impelling circumstances.

Though the attraction of the result reached is strong, I am unable to join the opinion of my brother in dissent. In this regard, I am in accord with the remarks of my brother in concurrence. It is an undeniable truism that Congress, along with this court and every other respecter of our patent system, intends that patents be issued only upon those claimed inventions which meet established standards of novelty, utility and obviousness. But a determination of whether claims meet the unobviousness standard requires that there be something over which the claims may or may not be held obvious. The troubling question may be stated as "Compared to what?" My primary difficulty with the board's decision, and with the otherwise consistent reasoning of the dissent, lies in their underlying premise, i.e., that the lost interference counts herein exist as comparison material useable in considering patentability of the claims on appeal. If they do not, it cannot be said that appellants' claims are obvious variants of anything. In my view, the subject matter of the lost counts was not available for com-

parison. It was not available under 35 USC 102 because it was not an invention made in this country. It was not available under 35 USC 135(a) because that section does not provide for comparison or any other testing of obviousness. That the broad congressional intent may have been to limit patent claims to those unobvious over prior inventions made in this country finds support in the enactment of 35 USC 102(g). I can find no support in the present statute, or in its legislative history, for the view that Congress intended the obviousness standard to be applied in the light of an invention set forth in the counts of an interference lost only because of an earlier foreign filing date.

If, upon return of the case, the examiner finds no other basis of rejection, adherence to the language of the statute in the present case may produce a clearly undesirable result, i.e., the existence of a second patent containing claims which vary only obviously from claims in an earlier patent. That result does, however, have at least one potential compensation. Adherence to the statute may encourage limited statutory amendment, applicable to the precise facts here confronted.

Until such time as it shall be provided in law that one who loses an interference because of an earlier foreign filing date of another shall thereafter be precluded from obtaining a patent on an invention which would have been obvious if the subject matter of the counts of that interference had existed in this country at the time the invention was made, we must reach the result herein when presented with the precise facts now before us.

Rich, Judge, concurring.

The purpose of these brief remarks is to preclude any upsetting effect the dissenting opinion might have on the court's total rejection of the solicitor's novel use of 35 USC 135(a) to create "prior art" for the purposes of 35 USC 103 when there is admittedly no other statutory basis to do so.

At the outset, I would emphasize that there clearly is no rejection before us other than a §103 obviousness rejection. Review of the record and the recording of the oral argument of the solicitor makes that amply clear. He categorically stated that the basis of rejection is §103. His use of §135(a) was solely for the purpose of providing the essential "prior art" under §103, against which the claimed invention can be measured for obviousness or unobviousness.

The dissenting opinion acknowledges that "the majority opinion correctly \* \* \* disposes of a 35 USC 103 rejection \* \* \*". This is an implicit rejection of the solicitor's

theory. The court is, therefore, unanimous in rejecting that theory.

In attempting to use §135(a) as the basis for affirming the board, the dissent is thus not applying the solicitor's novel theory but another novel theory that §135(a) gives rise to an "implied bar to appellants' claims arising from an adverse award of priority." (Emphasis mine.) No such "bar," implied or otherwise, has been argued to the court. It was not the Patent and Trademark Office that conceived of using an 1836 Senate report, dealing with conditions long since dissipated by the creation and effective operation of a Patent Office having an examining corps, to support a wholly new interpretation of the Patent Act of 1952.

The dissenting opinion undertakes to find its "implied bar," which was never recognized by the solicitor, only in an ambiguous sentence in the board's opinion. The board, attempting to rephrase the basis of the examiner's rejection said, somewhat ineptly:

As we interpret the reasoning of the examiner, it is simply that the subject matter sought to be patented is barred to appellant [sic] by reason of the award of priority as to the counts of the interference under 35 USC 135.

This is the one and only reference to §135 in the Office. No reference to it appears in the examiner's final rejection or in his subsequent action, or in the Examiner's Answer.

What did the board mean by "under 35 USC 135?" What does that clause modify? The dissent reads it, mistakenly, I think, as saying "barred \* \* \* under 35 USC 135." I think all the board was saying was "award of priority \* \* \* under 35 USC 135." Section 135 is the obvious statutory basis for awards of priority, and the latter reading makes sense. In the total absence of any indication by the board that it was predicating the "bar" on §135, rather than on the "award of priority," the former reading is, to say the least, somewhat strained. In any case it is a weak reed on which to lean in charging the majority with resting its decision on a "false premise" and with reaching "an absurd result." The board's statement is not quite as "specifically and plainly stated" as the dissent assumes it to be, at least to this reader. When the board wants to plainly and specifically make rejections *additive* to those of the examiner, it is quite able to do so under 37 CFR 1.196(b). This it did not do.

Needless to say, I agree with Judge Lane's opinion.

Miller, Judge, dissenting.

### Basic Errors in Majority Opinion

The majority and concurring opinions rest on a false premise, namely:

The sole basis of rejection is under 35 USC 103, the rejected claims being held obvious in view of the subject matter of the counts of the interference.

From this, an erroneous statement of the "sole issue" is then postulated, to wit:

whether claims may be rejected under 35 USC 103 on the ground that a losing party to an interference is not entitled to claims which are *asserted* to be obvious variations of the invention defined in the counts, when section 102(g) and interference estoppel are not applicable. [Emphasis supplied.]

The involved claims do not come before this court on a basis that they are *asserted* to be obvious. They have been *held* by the Board of Appeals to be not patentably distinct from the invention defined in the counts lost in the interference, and appellants' failure below even to argue the point should remove it from this court's review. In any event, the majority opinion assumes the correctness of the board's holding. As the board said:

It is our position that it would [sic] *prima facie* obvious to one of ordinary skill in this art to use the acid, the ester or the metal salt, interchangeably. Also, we consider that the use of adjacent homologs as the main chain or as the ester forming alcohol constitute [sic] obvious variations and are not patentably distinguishable from the counts. We note that not only have appellants not come forth with any showing of unobvious results, but have not even argued the question of obviousness.

The examiner consistently took the position, as stated in his answer, that:

Claims 6-7, 10-18 and 21-25 are rejected "under 35 USC 103" as unpatentable over the counts of Interference No. 97,329.

However, the board clearly and deliberately avoided committing itself to that ground of rejection. This is what it said:

Claims 6, 7, 10-18 and 21-25 stand rejected "under 35 USC 103" as unpatentable over the counts of Interference No. 97,329 in which a decision on priority adverse to appellants was rendered. *As we interpret the reasoning of the examiner, it is simply that the subject matter sought to be patented is barred to appellant [sic] by reason of*

the award of priority as to the counts of the interference under 35 USC 135: [Emphasis supplied.]

After specifically indicating that its rejection was not under 35 USC 102(g), the board made the following statement which, contrary to Judge Rich's concurring opinion, can hardly be said to represent a "total absence" of a section 135 basis for rejection:

Rather, the rejection is on the basis that appellant [sic] has lost the interference and, in view of the adverse decision on priority, is not entitled to claims which correspond to or are obvious variations of the invention as defined in the counts of the interference.

It is to be noted that the solicitor, in his brief (p. 3), quotes this statement. If the board had been relying solely on a 35 USC 103 rejection, it would have cited the *claims* of the Maltha et al. patent rather than the *counts*, which had been lost in an interference under 35 USC 135. Contrary to the impression that might be received from Judge Rich's concurring opinion, this court does not review the solicitor's interpretation of the board's opinion, but reviews the decision of the board. *Hunt v. Treppschuh*, 523 F.2d 1386, 1388, 187 USPQ 426, 428 (CCPA 1975).

Perhaps, as the majority opinion says, "the examiner's position was not specifically reversed by the board," but it should be clear from the foregoing that there was no *sole* basis of rejection under 35 USC 103. Accordingly, while the majority opinion correctly, albeit laboriously, disposes of a 35 USC 103 rejection, it fails to respond to the rejection specifically and plainly stated by the board, quoted above. An identical fact situation has been before the court in at least four previous cases. *In re Hilmer*, 57 CCPA 985, 424 F.2d 1108, 165 USPQ 255 (1970) ("Hilmer II"); *In re Risse*, 54 CCPA 1495, 378 F.2d 948, 154 USPQ 1 (1967); *In re Walker*, 41 CCPA 913, 213 F.2d 332, 102 USPQ 79 (1954); *In re Normann*, 32 CCPA 1248, 150 F.2d 708, 66 USPQ 308 (1945). Thus, there can be no question that the board means it when it states that the loser of an interference is not entitled to claims that are obvious variations of the invention defined in the counts. The court has a duty to respond to this statement directly.

### The Controlling Issue

The *controlling issue* in this appeal is whether appellants, having lost an interference on the basis of the foreign filing date of a patentee, are barred from ob-

taining claims that are "obvious variations" (not patentably distinct) of the invention defined in the counts of the interference.

### Legislative History and Congressional Intent

The interference was declared and decided pursuant to 35 USC 135, subsection (a), of which provides:

Whenever an application is made for a patent which, in the opinion of the Commissioner, would interfere with any pending application, or with any unexpired patent, he shall give notice thereof to the applicants, or applicant and patentee, as the case may be. The question of priority of invention shall be determined by a board of patent interferences (consisting of three examiners of interferences) whose decision, if adverse to the claim of an applicant, shall constitute the final refusal by the Patent and Trademark Office of the claims involved, and the Commissioner may issue a patent to the applicant who is adjudged the prior inventor \* \* \*. [Emphasis supplied.]

The original precursor of section 135 was section 8 of the Act of July 4, 1836 (5 Stat. 120-21), which provided:

That whenever an application shall be made for a patent which, in the opinion of the Commissioner, would interfere with any other patent for which an application may be pending, or with any unexpired patent which shall have been granted, it shall be the duty of the Commissioner to give notice thereof to such applicants, or patentees, as the case may be; and if either shall be dissatisfied with the decision of the Commissioner on the question of priority of right or invention, on a hearing thereof, he may appeal from such decision, on the like terms and conditions as are provided in the preceding section of this act; and the like proceedings shall be had, to determine which or *whether either of the applicants* is entitled to receive a patent as prayed for. [Emphasis supplied.]

Section 6 of the 1836 Act provided:

That any person or persons, having discovered or invented any new and useful art, machine, manufacture, or composition of matter, or any new and useful improvement on any art, machine, manufacture, or composition of matter, not known or used by others before his or their discovery or invention thereof, and not, at the time of his application for a patent, in public use or on sale, with his consent or allowance, as the inventor or discoverer; and shall desire to obtain an exclusive

property therein, may make application in writing to the Commissioner of Patents, expressing such desire, and the Commissioner, on due proceedings had, may grant a patent therefor \* \* \*.

Necessarily, the determination under section 8 of whether either of the applicants was entitled to receive a patent would have had to be consistent with the requirements for patentability prescribed by section 6.

The report of the Senate Select Committee on the State and Condition of the Patent Office (S. Rep. No. 338, 24th Cong., 1st Sess. 2-6 (1836)), which accompanied the bill that became the 1836 Act, said:

The Act of 1793, which is still in force, gives \* \* \* no power to the Secretary [of State] to refuse a patent for want of either novelty or usefulness. The only inquiry is whether the terms and forms prescribed are complied with \* \* \*. The necessary consequence is, that patents have \* \* \* been daily granted without regard to the question of novelty, or even utility in the ordinary sense \* \* \*.

Under the act referred to, the Department of State has been going on for more than forty years, issuing patents on every application, without any examination into the merit or novelty of the invention. And the evils which necessarily result from the law as it now exists, must continue to increase and multiply daily till Congress shall put a stop to them. Some of them are as follows:

\* \* \*

4. It opens the door to frauds, which have already become extensive and serious. It is represented to the committee that it is not uncommon for persons to copy patented machines in the model-room; and, having made some slight *immaterial alterations*, they apply in the next room for patents \* \* \*.

In this collision and *interference* of patents, the original and meritorious inventor sees his invention, to the perfection of which he has devoted much time and expense, pirated from him, and he must forego the reward which the law was intended to secure to him in the exclusive right it grants; or he must become involved in numerous and expensive lawsuits in distant and various sections of the country, to protect and confirm his rights. [Emphasis supplied.]

Declaring that "the first and most desirable object of a revision and alteration of the existing laws on this subject" was to prevent the evils which had resulted from "the un-

restrained and promiscuous grants of patent privileges," the report continued:

A power in the Commissioner of the Patent Office to reject applications for want of novelty in the invention, it is believed, will have a most beneficial and salutary effect in relieving meritorious inventors, and the community generally, from the serious evils growing out of the granting of patents for every thing indiscriminately, *creating interfering claims*, encouraging fraudulent speculators in patent rights, deluging the country with worthless monopolies, and laying the foundation for endless litigation.

\* \* \*

\* \* \* The present law waits till infringements and frauds are consummated — nay, it even aids them; and then it offers an inadequate remedy for the injury, by giving an action for damages. It ought, rather, *by refusing to grant interfering patents*, to render prosecutions unnecessary. [Emphasis supplied.]

From the foregoing, it is clear that the intent of Congress in enacting section 8 of the 1836 Act was not only to prevent the granting of interfering patents but also to prevent an applicant involved in an interference from obtaining a patent unless his invention satisfied the requirements for patentability. See *Jones v. Wetherill*, 13 F. Cas. 1069 (No. 7,508) (C.C.D.C. 1855); *Potter v. Dixon*, 19 F. Cas. 1145 (No. 11,325) (C.C.S.D.N.Y. 1863).

There is nothing in the amendatory language or legislative history of subsequent statutes, including the Act of July 8, 1870, ch. 230, 16 Stat. 198, 204, and the Act of July 19, 1952, ch. 950, Pub. L. No. 593, 66 Stat. 792, 801-02, to show any change in this Congressional intent. Although the "whether" provision in section 8 of the 1836 Act was dropped (without legislative comment) from the successor section (§42) of the 1870 Act, Rule 59 of the Patent Office, promulgated July 15, 1870, in accordance with the 1870 Act provided:

<sup>1</sup> Under the Patent Act of 1790 (1 Stat. 109), the Secretary of State examined applications for patentability. However, examination was eliminated by the Patent Act of 1793 (1 Stat. 318) and not restored until the 1836 Act. It was the examination system, which has been expanded and refined over the ensuing one hundred and forty years, which insured that the patent grant would not be "unrestrained and promiscuous."



Act of 1790 (1 Stat. 109), examined applications for every examination was in the Act of 1793 (1 Stat. 318) and the 1836 Act. It was the examination which has been expanded and now covers one hundred and forty thousand of the patent grant would be "promiscuous."

<sup>2</sup> It is to be noted that Rule 232 of the Patent Office Rules of Practice, March 1, 1949, and January 1, 1953, also provided for unpatentability of the counts to an applicant involved in an interference with another applicant as a basis for a motion to dissolve the interference. Such long-continued administrative interpretation is a presumptively correct interpretation of the statute involved. *Costanzo v. Tillinghast*, 287 U.S. 341, 345 (1932); *National Lead Co. v. United States*, 252 U.S. 140, 145-46 (1920).

<sup>5</sup> In *Barrett v. Hall*, 2 F.Cas. 914, 923 (No. 1047) (C.C.D. Mass. 1818), Justice Story said: "The true legal meaning of the principle of a machine, with reference to the patent act, is the peculiar structure or constituent parts of such machine."

Thus, in *Whitney v. Emmett*, 29 F. Cas. 1074, 1078 (No. 17,585) (C.C.E.D. Pa. 1831), Justice Baldwin declared:

The novelty of an invention is either the manufacture produced, or the manner of producing an old one; if the patent is for the former, it must be for something substantially new, different from what was before known; if the latter, the mode of operation must be different, not a mere change of the form and proportions; if both are the same in principle, structure, mode of operation, and produce the same result, they are not new, though there may be a *variance* in some small matter for the purpose of evasion, or as a colour for a patent." [Emphasis supplied.]

In a similar vein, Curtis, Law of Patents 6-7 (1849) states:

So, too, mere *colorable variations*, or slight and unimportant changes, will not support a patent \* \* \*. In such cases, if the consequences resulting from the change are unimportant, and the change consists merely in the employment of an *obvious* substitute, the discovery and application of which could not have involved the exercise of the inventive faculty, in any considerable degree, then the change is treated as merely a *colorable variation*, or a double use, and not as a substantive invention. [Emphasis supplied.]

Webster, Law and Practice of Letters Patent for Inventions 49 (London 1841) puts it as follows:

He is not the true and first inventor who has a material part of the invention suggested to him by another \* \* \*.

A similar approach was taken in defining the concept of "identity" of invention. Absolute identity was not required. Curtis, *supra* at 262, says:

An infringement involves substantial identity, whether that identity is described by the terms, "same principle," same *modus operandi*, or any other. It is a copy of the thing described in the specification of the patentee, either without variation, or with only such *variations* as are consistent with its being in substance the same thing. [Emphasis supplied.]

*Substantial* identity was determined to be the test in an interference in *Tyson v. Rankin*, 24 F. Cas. 490, 493 (No. 14,320) (C.C.D.C. 1853).

It fell to the Supreme Court to break with the preoccupation over "principle" and "substance" of the invention. In *Hotchkiss v. Greenwood*, 52 U.S. 248 (1850), the

Court, deciding a case under the 1836 Act, declared:

For unless more ingenuity and skill \* \* \* were required \* \* \* than were possessed by an ordinary mechanic acquainted with the business, there was an absence of the degree of skill and ingenuity which constitute essential elements of every invention. In other words, the improvement is the work of the skilful mechanic, not that of the inventor.

Thus, a patentable invention requires more ingenuity and skill than that possessed by a routinier — a person having ordinary skill in the art to which the subject matter pertains — namely, "unobviousness."<sup>6</sup>

In view of the foregoing, the following conclusion is inescapable: If we are to give meaningful recognition to the intent of Congress, carried down through the years from the Act of 1836, that an applicant involved in an interference proceeding shall not obtain a patent unless his invention satisfies the requirements for patentability, then section 135, *quite apart from section 103 and section 102(g)*, must at least be read to bar, by implication, an applicant who has lost an interference on the basis of the foreign filing date of a patentee from obtaining claims for not only the invention defined by the interference counts but also for any invention representing variations requiring no more ingenuity and skill than that possessed by a person having ordinary skill in the art to which the interference counts pertain (i.e. what the board termed "obvious variations"). As well stated by the board:

If we were to follow appellants' reasoning to its logical conclusion, we would condone a situation in which, by presenting claims, drawn sequentially, to com-

<sup>6</sup> In *I. Robinson*, Law of Patents (1890) 305-06, the author emphasized the need for substantial variation for novelty to exist as follows:

Every invention which is not already accessible to the public is regarded in law as new to the public \* \* \*. Novelty, therefore, exists unless the invention is already in the possession of the public as an operative art or instrument, and this occurs only when the invention is a matter of existing public knowledge, or is derivable from what is known without further exercise of inventive skill. In other words, as every variation of form, as distinguished from variation in substance, is considered as effected by the imitative faculties, novelty consists in the substantial variation of the invention in question from all inventions which in contemplation of law are already open to the public.

under the 1836 Act,

... and skill \* \* \*  
... than were possessed  
... acquainted with  
... an absence of the  
... ingenuity which con-  
... of every inven-  
... the improvement is  
... mechanic, not that

... ion requires more  
... that possessed by a  
... ing ordinary skill  
... subject matter per-  
... viousness."<sup>6</sup>

... ing, the following  
... If we are to give  
... to the intent of  
... through the years  
... hat an applicant in-  
... ce proceeding shall  
... less his invention  
... ts for patentability,  
... t from section 103  
... at least be read to  
... applicant who has  
... on the basis of the  
... patentee from ob-  
... only the invention  
... ce counts but also  
... resenting variations  
... uity and skill than  
... on having ordinary  
... the interference  
... the board termed  
... as well stated by the

... appellants' reason-  
... elusion, we would  
... which, by present-  
... quentially, to com-

Patents (1890) 305-06,  
... need for substantial  
... is follows:

... not already accessible  
... in law as new to the  
... therefore, exists unless  
... in the possession of the  
... or instrument, and  
... invention is a matter  
... age, or is derivable  
... out further exercise of  
... words, as every varia-  
... ied from variation in  
... as effected by the im-  
... onists in the substan-  
... tion in question from  
... contemplation of law  
... public.

pounds containing carbon chains from 1 to 20 carbon atoms, or to obvious variations of the compounds, the losing party in an interference with a patent (which relied on a foreign priority date) could be granted a patent which would cover all aspects of the invention defined in the counts of the interference as to which he has been adjudicated not to be the first inventor. Obviously, this would make a mockery of the interference practice.

To this it might be added that intent for such an absurd result is not to be attributed to the Congress. *Haggar Co. v. Helvering*, 308 U.S. 389, 394 (1940).

### Other Errors in Majority Opinion

Instead of following basic rules of statutory construction that would lead to a correct decision, the majority opinion reaches an absurd result and then says that Congress should change the law.<sup>7</sup> It ignores the legislative history of section 135 and the clearly *substantive* intent of Congress that an applicant involved in an interference proceeding shall not obtain a patent unless his invention satisfies the requirements for patentability. Instead, it simply says:

A reading of section 135(a) confirms that all its provisions relate to procedure rather than substance.

The majority opinion cites the Commentary on the New Patent Act, 35 USCA at 37-38, for the statement that sections 131-135 deal with "procedure." But it ignores the fact that the Commentary does *not* say that these sections deal *only* with procedure and, in

commenting on the provision in section 135 for cancellation of claims involved in an interference, states:

This is new *in substance* and is made possible by the amplification of the right of review of the patentee provided for in section 146. [Emphasis supplied.]

Other provisions, such as new matter (section 132), the time bar (section 135(b)), and unenforceability (section 135(c)), are clearly substantive.

Instead of reviewing the board's specific ground of rejection (which is set forth in appellants' reason of appeal No. 3), the majority opinion concentrates on rebutting the solicitor's novel proposition that 35 USC 135 is a prior art statute. But it would be a non sequitur to conclude that this precludes reliance on section 135 for an implied bar to appellants' claims arising from an adverse award of priority.<sup>8</sup>

The absurdity of the result reached by the majority opinion is pointed up by its discussion of this court's opinion in *In re Ogive*, 517 F.2d 1382, 186 USPQ 227 (CCPA

\* Appellants contend that an award of priority does not per se preclude the losing party from allowance of claims to subject matter not adjudicated in the interference, citing *In re Frilette*, 58 CCPA 799, 436 F.2d 496, 168 USPQ 368 (1971) and *In re Taub*, 52 CCPA 1675, 348 F.2d 556, 146 USPQ 384 (1965). Although appellants' "per se" contention is correct, as those cases demonstrate, the cases do not help appellants. In *Frilette*, the court disagreed with the solicitor's position that the claims involved there must be patentably distinct from the interference counts, because the interference decision did not show who was the first inventor of the "subject matter" and appellants had been prevented from making such a showing in the interference. Appellants' motion to broaden the interference counts to permit them to establish their earliest date of priority had been denied. The court held that appellants could not be denied the opportunity to show by affidavit that their act of inventing the subject matter of claims identical to the broadened counts proposed by the motion was prior to that of the patentee. That is not the situation here, where appellants neither moved to broaden the interference counts nor attempted to antedate Maltha et al.'s date of invention. In *Taub*, there had been an award of priority on a broad count based on evidence of priority with respect to only one species. The court pointed out that domination by the count did not preclude a showing of patentable distinction with respect to another species by a party whose invention of that species was prior to the invention of the interference winner. Clearly that case is inapposite. The decisions in both cases are entirely consistent with the Congressional intent under 35 USC 135 referred to earlier in this opinion.

So does Chief Judge Markey's concurring opinion. The rule that this court developed in dealing with obviousness-type double patenting in cases involving obvious variations of an invention claimed in an earlier patent would never have come about if the "leave it to Congress" attitude had prevailed. That rule represents recognition of what this court characterized as "one of the fundamental principles underlying the patent system, namely, that when the right to exclude granted by a patent expires at the end of the patent term, the public shall be free to use the invention as well as obvious modifications thereof or obvious improvements thereon." *In re Robeson*, 51 CCPA 1271, 1275, 331 F.2d 610, 614, 141 USPQ 485, 488 (1964). The court added that "to grant a second patent for an obvious variation deprives the public of those rights." Such deprivation of the public would be no less if the loser in an interference were later granted a patent for obvious variations of the invention of the counts.

1975). It proposes to draw an artificial distinction between a concession of priority (arising from a party's refusal to copy claims which he could make) that prevents an interference and a concession of priority in an interference that results in an award of priority. In *Ogiue*, the applicant apparently refused to copy the claims because he would lose the interference based on his opponent's foreign priority date. Under the decision reached by the majority here, however, *Ogiue* could have been assured of his claims (to what the court there said was essentially the same invention as that of the patentees) by simply copying the claims of the patentees and forcing them to prove right to priority based on their foreign priority date. In the applicant's subsequent ex parte proceeding, the record of the interference would show that the patentees had made the invention outside the United States, while the applicant would be treated as the first to make the invention in the United States.<sup>9</sup> The patentees, having won the interference, would be in a worse position than if there had been no interference. This is the situation the board here had in mind when it said:

Obviously, this would make a mockery of the interference practice.

#### Normann and Walker Overruled

Finally, the majority and the concurring opinions lead the court to overrule *In re Normann*, supra, and *In re Walker*, 41 CCPA 913, 213 F.2d 332, 102 USPQ 79 (1954). In each case, the appellant had lost an interference based on the foreign priority date of his opponent. The principal issue in *Normann* was "whether the use of fresh acid in the claimed process involves invention over the counts of the interference, which counts \* \* \* call for the use of acid broadly." This court held that the limitation of "freshness" did not involve "invention" over the lost counts and affirmed the rejection by the board. The opinion of the court did not refer to "prior art" and did not men-

tion *res judicata*<sup>10</sup> or estoppel. It clearly supports the Congressional intent that an applicant involved in an interference proceeding shall not obtain a patent unless his invention satisfies the requirements for patentability. Presumably the law of this case was carried over into the Act of July 19, 1952, one of the objectives of which was "the codification of the patent statutes and some prior case law." Commentary on the New Patent Act, 35 USCA at 10.<sup>11</sup> The majority and concurring opinions are barren of any showing to the contrary. As pointed out earlier, there is nothing in the amendatory language or the legislative history of the 1952 Act to show any change in the above-referred-to Congressional intent.

Similarly, the opinion in *Walker* did not refer to "prior art" and did not mention *res judicata* or estoppel. Just like the board here, it relied not at all on 35 USC 102(g), and it is significant that Chief Judge Markey's concurring opinion does not say that this court in *Walker* "had to have done just that." It merely noted the solicitor's argument that the court had uniformly applied the rule that "a losing party in an interference is not entitled to claims that would dominate the claims given to his successful rival."<sup>12</sup> Although the appealed claims here, if allowed, would not "dominate" those awarded *Maltha et al.* in the interference, like those in *Walker* they

<sup>10</sup> The U.S. Court of Appeals for the District of Columbia applied *res judicata* against the losing party in an interference in *United States Rubber Co. v. Coe*, 146 F.2d 315, 64 USPQ 100 (1945).

<sup>11</sup> See Judge Baldwin's concurring opinion in *In re Bass*, 59 CCPA 1342, 1375 and 1377, 474 F.2d 1276, 1302 and 1303, 177 USPQ 178, 197 and 198; also the main opinion at 59 CCPA 1359, 474 F.2d 1289, 177 USPQ 188.

<sup>12</sup> The court cited *In re Sola*, 22 CCPA 1313, 77 F.2d 627, 25 USPQ 433 (1935), where appellant had conceded priority of invention in an interference and the court held that the claims on appeal contained limitations which, when considered with the other elements of the claims, did not define anything inventive over the invention on which priority had been conceded; and *In re Derleth*, 28 CCPA 973, 118 F.2d 566, 49 USPQ 84 (1941), where the court held appellant was estopped from making claims which it found were not patentably distinct from the counts of the interference and appellant had not objected to the holding of the Primary Examiner that the claims would be held subject to the outcome of the interference. But see *In re Long*, 23 CCPA 1078, 83 F.2d 458, 29 USPQ 357 (1936), where dominating claims were allowed to the losing party of an interference since the record disclosed that all parties to the interference other than appellant clearly conceded that they could not be regarded as the first inventor of the subject matter of the dominating claims.

<sup>9</sup> If, in fact, the party with the earlier foreign priority date had made the invention in the United States before the losing party's effective United States date, the record would not necessarily show it, because, in the interference, all the winning party would have had to show was his foreign priority date. For all the record here shows, *Maltha et al.* might have made the invention of the counts in the United States before appellants' date of June 6, 1963, since they needed to show only their right to their Netherlands filing date to be awarded priority. See *In re Ogiue*, supra at 1391 n.3, 186 USPQ at 235 n.3.

## District Court, N. D. California

Crumb v. A. A. Sales, Inc.

No. C-73-2103 Decided Sept. 3, 1975

## COPYRIGHTS

## 1. Notice of copyright (§24.35)

Pleading and practice in courts —  
Motions — For summary judgment  
— In general (§53.6331)

Accused infringer is entitled to summary judgment if its allegations, that copyright owner consented to use of copyrighted cartoon character on business cards and that thousands were distributed without 17 U.S.C. 10 copyright notice, are not controverted.

## 2. Notice of copyright (§24.35)

Publication of copyrighted cartoon character without copyright notice on thousands of business cards circulated among public, without claim that cartoon was used to bring copyright owner's product to select buying group's attention, is dedication to public that ends copyright protection.

## 3. Affidavits — In general (§12.1)

Pleading and practice in courts —  
Motions — For summary judgment  
— In general (§53.6331)

Accused infringer's evidence that copyrighted figure was published without copyright notice, with copyright owner's permission, is not controverted by copyright owner's affidavit that his present state of mind was that he had given no such information, leading court to grant accused infringer's motion for summary judgment.

Action by Robert Crumb against A. A. Sales, Inc., for copyright infringement. On defendant's motion for summary judgment. Motion granted.

See also 188 USPQ 447.

Albert L. Morse, Michael Stepanian, and Michael L. Krassner, all of San Francisco, Calif., for plaintiff.

Naylor, Neal & Uilkema, and John K. Uilkema, both of San Francisco, Calif., and Ford E. Smith, Seattle, Wash., for defendant.

Wollenberg, District Judge.

This is an action to enforce Plaintiff's copyright of a cartoon figure. In this motion for summary judgment, Defendant claims the Plaintiff consented to the publication of

do not "define subject matter which [is] patentably distinct."<sup>1</sup> In Walker, this court expressly stated that the rejection was *not* based on the interference winner's foreign priority date,<sup>2</sup> but "upon the adverse award of priority of invention against appellant." Thus, Walker is squarely in point and clearly supports the Congressional intent that an applicant involved in an interference proceeding shall not obtain a patent unless his invention satisfies the requirements for patentability. The court in Walker and Normann did not give the interference winner a Pyrrhic victory by saying, "Let Congress change the law." Why should it do so today?

## Conclusion

Lurking behind the majority's decision is the specter of *In re Hilmer*, 57 CCPA 985, 424 F.2d 1108, 165 USPQ 255 (1970) ("Hilmer II"). The issue upon which the decision in Hilmer II was narrowly focused, however, is *not* before us, either for reaffirming or overruling, because the board expressly disavowed reliance on 35 USC 102(g) coupled with 35 USC 119. The issue that is before us is whether appellants, having lost an interference on the basis of the foreign filing date of Maltha et al., are barred from obtaining claims that are obvious variations of the invention defined in the counts lost in the interference.

In view of the foregoing, the court should hold that they are.

<sup>1</sup> Although subservient claims may, if they represent a patentably distinct invention, be patentable, they cannot be so regarded if they are merely obvious variations of the dominant claim. *In re Taub*, supra note 8.

<sup>2</sup> Thereby the court avoided the "push back" argument which had been rejected in *In re Hilmer*, 53 CCPA 1288, 359 F.2d 859, 149 USPQ 480 (1966) ("Hilmer (I)"), just as the board did here.

ion in Walker did not and did not mention res just like the board all on 35 USC 102(g), that Chief Judge opinion does not say that had to have done noted the solicitor's court had uniformly losing party in entitled to claims that claims given to his through the appealed allowed, would not Maltha et al. in those in Walker they

seals for the District of data against the losing in United States Rubber 100 USPQ 100 (1945). concurring opinion in 42, 1375 and 1377, 474 303, 177 USPQ 178, 197 at 59 CCPA 1359, 188. Sola, 22 CCPA 1313, 77 (1935), where appellant invention in an in held that the claims on ations which, when con- of the claims, did over the invention been conceded; and *In re* 18 F.2d 566, 49 USPQ appellant was es- which it found were from the counts of the in- and not objected to the Examiner that the claims to the outcome of the in- long, 23 CCPA 1078, 83 (1936), where dominat- to the losing party of an record disclosed that all other than appellant could not be regarded the subject matter of the

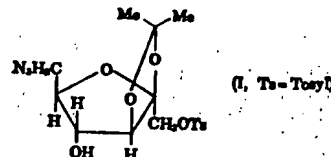
from trifluoroacetates,  $\text{CF}_3$ . The pyr. li. is characteris-  
tic for the acetates and trifluoroacetates. The decomposition of  
acetates is the decompn. of two groups with the elimination of  
acetic acid and  $\text{Ac}_2\text{O}$ . Trifluoroacetates do not behave thus, but  
eliminate  $\text{CF}_3\text{CO}_2\text{H}$ . The decompn. of 1,6-acetimidino sugars  
follows a different route, leading to a stable end product which  
remains the intact ring.  
D. H. Hutson

65779t Thin-layer chromatography of carbohydrates. A.  
Pastuszyn and H. Michl. *Mitt. Versuchslab. Gaerungsgewerbe*  
20(1-2), 1-4 (1966)(Ger). To sep. and detect 0.04-0.08  
mole ams. of carbohydrates by thin-layer chromatog., develop  
the mixt. on Silica Gel G twice in the same direction with  $\text{BuOH}$ -  
 $\text{EtOH}$ - $\text{H}_2\text{O}$  (5:1:4), drying between runs, then once transversely  
with  $\text{BuOH}$ : $\text{HOAc}$ : $\text{Et}_2\text{O}$ : $\text{H}_2\text{O}$  (9:6:3:1). Let the solvent front  
move 14 cm. each time. Spray 1st with 1:2 2N  $\text{PhNH}_2$  in  
 $\text{BuOH}$ -2N  $\text{H}_3\text{PO}_4$ ; heat 10 min. at  $110^\circ$  to detect almost all  
compds. by uv fluorescence. Spray again with 10:1 0.4% orcinol  
in  $\text{EtOH}$ -conc.  $\text{H}_3\text{PO}_4$ ; heat as before to detect ketoses, sucrose,  
and raffinose. Spray a 3rd time with 10:1 0.4% naphthoresorcinol  
in  $\text{EtOH}$ :conc.  $\text{H}_3\text{PO}_4$ ; heat as before to detect aldopentoses,  
aldohexoses, ketoses, and other sugars as yellow-brown spots.  
Development time totals 7.5 hrs. C<sub>4</sub>-4 compds. cannot be sepd.  
because of the reaction with silica gel. Oligosaccharides such as  
maltotriose and maltohexose are sepd. better with  $\text{BuOH}$ :iso-  
 $\text{PrOH}$ : $\text{H}_2\text{O}$  mixts. Pictorial diagrams are shown for the sepn. and  
detection of arabinose, 2-deoxy-arabino-hexose, 2-deoxy-erythro-  
pentose, fructose, fucose, galactose, 2-amino-2-deoxyglucose,  
glucose, lactose, lyxose, maltose, mannose, 2-acetamido-2-de-  
oxyglucose, raffinose, rhamnose, ribose, sucrose, sorbose, taga-  
nose, trehalose, xylose, glucuronic acid, maltotetraose, melibiose,  
galactobiose, "dendroketo" (I), digitoxose, 5-hydroxymethyl-  
furfural (II), galacturonic acid, maltotriose, and melezitose in  
synthetic mixts., in wine, and in the condensation product of  
 $\text{HCHO}$  (III). Prep. I by treating 1 g.  $\text{CO}(\text{CH}_2\text{OH})_2$  with 20  
ml. of 0.5N  $\text{NaOH}$  at room temp. for 4 hrs. Neutralize with  
conc.  $\text{HCl}$ , steam-distill, and ext. the residue with 80%  $\text{EtOH}$ .  
Disc. the ext., then sep. I by paper chromatog. Prep.  
II by heating 10 g. sucrose with 30 ml. of aq. 0.3%  $(\text{COOH})_2$   
in a closed vessel at  $145^\circ$  for 15 min., then at  $125^\circ$  for 2 1/2 hrs.  
Cool, neutralize with  $\text{CaCO}_3$ , and ppt. impurities with 0.5 g.  
 $\text{NaOAc}$ . Ext. with  $\text{EtOAc}$ , dry over anhyd.  $\text{MgSO}_4$ , distil  
the solvent, and crystallize II (m.p.  $31^\circ$ ) from the brown sirup.  
Prep. III by heating 0.1M  $\text{HCHO}$  in aq. 0.04N  $\text{LiOH}$  and 0.001N  
 $\text{NaOH}$ , for 14 min. Cool quickly, desalt with Dowex 50, steam  
distill, dissolve in  $\text{MeOH}$ , and steam distill repeatedly to remove  
the residual  $\text{HCHO}$ .  
J. Bornstein

65780m Methylation of carbohydrates bearing base-labile  
substituents, with diazomethane-boron trifluoride etherate. I.  
O. Mastronardi, S. M. Flematti, J. O. Deferrari, and E. G.  
Garcia (Univ. Buenos Aires). *Carbohydr. Res.* 3(2), 177-83  
(1966)(Eng). Various tetra-O-acetyl-D-hexopyranoses undergo  
acetyl migration on methylation with Purdie's reagents; thus,  
1,3,4-tetra-O-acetyl- $\beta$ -D-glucopyranose, 1,2,3,6-tetra-O-acetyl- $\beta$ -  
glucopyranose, and 1,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranose  
all its anomers are converted into methyl 2,3,4,6-tetra-O-acetyl-  
glucopyranoside when methylated with  $\text{MeI}$  and  $\text{Ag}_2\text{O}$ .  
When the compd. is treated with  $\text{CH}_3\text{N}_2$  and  $\text{BF}_3 \cdot \text{Et}_2\text{O}$ , the  
methylated product is produced in a high yield, with the  $\text{Me}$  ether  
group in the expected position. Under the same conditions,  
1,4,6-tetra-O-acetyl- $\alpha$ -D-galactopyranose (I) is converted into  
methyl 2-O-Me ether without the acetyl migration observed when I  
is methylated with Purdie's reagents. The tetra-O-acetyl-O-  
methyl-D-hexopyranoses were transformed by deacetylation  
to the following mono-O-methylhexoses: 2-O-methyl-D-glucose,  
4-O-methyl-D-glucose, 6-O-methyl- $\alpha$ -D-glucose, and 2-O-  
methyl- $\beta$ -D-galactose. 16 references.  
RCYM

65781n Synthesis of hydropiperidines from carbohydrate  
precursors. Stephen Hanessian (Parke, Davis & Co., Ann  
Arbor, Mich.). *Chem. Ind. (London)* 1966(51), 2126-7(Eng).  
Redn. of 5-azido-5-deoxypentoses in  $\text{MeOH}$  in the presence of 6  
wt%  $\text{Pd/C}$  at room temp. and atm. pressure for 1-2 hrs. af-  
forded the corresponding 3,4,5-trihydroxypiperidines, charac-  
terized as the N-acetyl derivs., in a very good yield, e.g. N-acetyl-  
3,4,5-trihydroxypiperidine, m.  $193-4^\circ$ ,  $[\alpha]_D^{20} 0^\circ$  (c 3.10  
in  $\text{H}_2\text{O}$ ) in quant. yield. Reductive rearrangement of 5-azido-2,3-  
benzylidene-5-deoxy- $\beta$ -D-ribofuranose afforded after N-acetyla-  
tion, N-acetyl-3,4-O-benzylidene-D-ribo-3,4,5-trihydroxypiperi-  
dine, m.  $154-6^\circ$ ,  $[\alpha]_D^{25} -42.7^\circ$  (c 1.53  $\text{MeOH}$ ). 6-Azido-6-de-  
oxy-D-glucose, m.  $128-30^\circ$ , or the D-galacto analog, m.  $144-5^\circ$ ,  
after acetylation the corresponding 6-acetamido-6-deoxy-  
sugars rather than septanose products with N in the ring.  
Redn. of 6-azido-6-deoxy-D-gluconolactone, m.  $138-40^\circ$ , af-  
forded 6-amino-6-deoxy-D-gluconolactam, m.  $210-12^\circ$  (de-  
compn.). Reaction of 2,3-O-isopropylidene-1,6-di-O-p-tolyl-  
sulfonyl-D-fructofuranose with 1 molar equiv. of  $\text{NaN}_3$  in  $\text{HCONH}_2$   
at  $100^\circ$  for 18 hrs. gave 6-azido-6-deoxy-2,3-O-isopropyl-  
sulfonyl-1-O-p-tolylsulfonyl-D-fructofuranose (I), m.  $78-8^\circ$ . Re-  
arrangement of the p-tolylsulfonyloxy function in I by a benzoate  
subsequent debenzoylation and hydrolysis afforded 6-azido-  
6-deoxy-D-fructofuranose (II). Reductive rearrangement of II

gave a 2-hydroxymethyl-3,4,5-trihydroxypiperidine,  $[\alpha]_D^{25} -65^\circ$ ,  
(c 3  $\text{MeOH}$ ); N-acetyl deriv. m.  $198-0^\circ$ ,  $[\alpha]_D^{25} -11^\circ$  (c



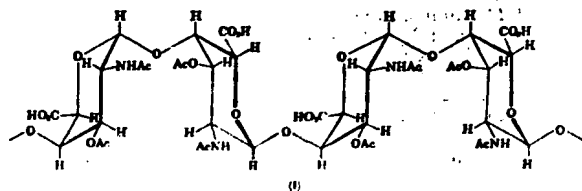
0.4  $\text{MeOH}$ ). Reductive rearrangement of 5-azido-5,6-dideoxy-  
L-talose and subsequent acetylation gave N-acetyl-6-methyl-L-  
talo-3,4,5-trihydroxypiperidine,  $[\alpha]_D^{25} 11.4^\circ$  (c 1.55  $\text{MeOH}$ ).  
A. Foxton

65782p Sequence studies on *Diplococcus pneumoniae* type II  
polysaccharide. S. A. Barker, P. J. Somers, and M. Stacey  
(Univ. Birmingham, Engl.). *Carbohydr. Res.* 3(3), 261-70  
(1967)(Eng). Assay of  $\text{HCO}_2\text{H}$  released by periodate oxidn. of  
*D. pneumoniae* Type II polysaccharide (SII), during liberation of  
an L-rhamnose-contg. trisaccharide from SII by  $\alpha$ -L-rhamno-  
sidase, demonstrated an  $\alpha$ -(1  $\rightarrow$  4) linkage between this trisac-  
charide and the remainder of the SII mol. Carboxyl-reduced  
SII,  $[\alpha]_D^{25} 42^\circ$ , contg. 49.7% D-glucose and 49.8% L-rhamnose  
was prepd. by redn. of the 2-hydroxyethyl ester in two stages.  
A comparison of the cleaved products of the action of specific,  
induced  $\alpha$ - and  $\beta$ -D-glucosidases on SII and carboxyl-reduced SII  
demonstrated the presence of O- $\beta$ -(D-glucopyranosylidronic acid)-  
(1  $\rightarrow$  4)-O- $\alpha$ -D-glucopyranosyl-(1  $\rightarrow$  4) residues in SII and O- $\beta$ -D-  
glucopyranosyl-(1  $\rightarrow$  4)-O- $\alpha$ -D-glucopyranosyl-(1  $\rightarrow$  4) residues in  
the reduced polysaccharide. A possible repeating unit for SII  
is discussed.  
RCYM

65783q Influence of the hydrogen ion concentration on the  
specificity of the TTC reaction. Klaus Lorenz and Hans Hoff-  
meister (Univ. Hamburg, Ger.). *Mikrochim. Acta* 166(6),  
1062-7(Ger). Triphenyltetrazolium chloride (TTC), is widely  
used as a dehydrogenation reagent for org. compds. The  $\text{H}_2\text{O}$ -  
sol. TTC salt is converted during this reaction into the corre-  
sponding formazan, a red insol. compd. The spontaneous forma-  
tion of formazan occurs only in alk. media and the number of  
compds. responding to dehydrogenation increases with increas-  
ing pH of the environment. The threshold pH of 12.45, above  
which TTC remains inactive, could be lowered to 12.0 by addn.  
of  $\text{MeOH}$  or *tert*-BuOH. This shift reduces the no. of reagent  
partners, increases the specificity of the dehydrogenation, and  
allows one to follow quant. the progress of reaction. Data on  
D-glucose, D-glucose 1-phosphate, sucrose, cellulose powder, sol.  
starch, glycogen, 2-amino-2-deoxy-D-glucose hydrochloride,  
chondroitin sulfate, hyaluronic acid, heparin, Na lactate, Na K  
tartrate, Na pyruvate,  $\alpha$ -oxoglutaric acid, cysteine hydro-  
chloride, creatinine, urea, and glycine are tabulated.  
G. W. Scherf

65784r Preparation and characterization of 2-O-(4-O-methyl-  
 $\alpha$ -D-glucopyranosyl)-D-xylopyranose. Nirmolendu Roy and T.  
E. Timell (State Univ., Coll. of Forestry, Syracuse, N.Y.).  
*Carbohydr. Res.* 3(2), 246-7(1966-1967)(Eng). 2-O-(4-O-Methyl-  
 $\alpha$ -D-glucopyranosyl)-D-xylopyranose, prepd. by  
partial acid hydrolysis of a (4-O-methylglucuronoxylan from  
aspen wood, was acetylated with  $\text{Ac}_2\text{O}$ -NaOAc. The product  
was reduced with  $\text{B}_2\text{H}_6$  in tetrahydrofuran and saponified with  
 $\text{NaOMe}$  to give 57% title compd., m.  $95-105^\circ$ ,  $[\alpha]_D^{25} 114^\circ$   
( $\text{H}_2\text{O}$ ).  
Derek Horton

65785s Elucidation of the structure of the Vi-antigen isolated  
from *Escherichia coli* 5396/38. K. Heyns and G. Kiessling  
(Univ. Hamburg, Ger.). *Carbohydr. Res.* 3(3), 340-53(1967)  
(Ger). The structure of the Vi-antigen isolated from *E. coli*  
5396/38 was elucidated by applying (1) methylation followed by  
redn., and (2) redn. prior to methylation. Hydrolyzates of the  
Vi-antigen material treated in these two ways contained 2-amino-  
deoxy-3-O-methyl-D-galactopyranose (from 1) and 2-amino-2-  
deoxy-3,6-di-O-methyl-D-galactopyranose (from 2), resp. The oc-  
currence of these 2 compds., as well as the absence of 2-amino-2-  
deoxy-4-O-methyl-D-galactopyranose and 2-amino-2-deoxy-4,6-  
di-O-methyl-D-galactopyranose, conclusively demonstrates 1  $\rightarrow$   
4 glycosidic bonds in the polysaccharide. The products formed  
by hydrolysis were quant. detd. and identified using authentic



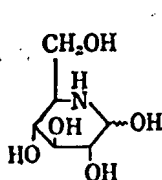
control substances in the amino acid analyzer of Moore, Spack-  
man, and Stein by a special buffer. From these results a model  
I of the structure of the Vi-antigen is presented. 20 references.  
RCYM

SACKI

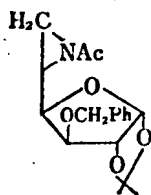
### Synthesis of Nojirimycin, D-Glucopiperidine

In the course of studies on 5,6-epimino carbohydrates,<sup>1)</sup> we have synthesized a monosaccharide antibiotic, nojirimycin (I), which was produced by some strains of *Streptomyces* and indicated activity against a drug-resistant strain of *Shigella flexneri*, *Sarcina lutea* and *Xanthomonas oryzae*.<sup>2)</sup>

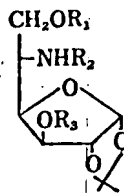
5,6-Acetylepimino-3-O-benzyl-5,6-dideoxy-1,2-O-isopropylidene- $\beta$ -L-idofuranose<sup>1)</sup> (II) was treated with acetic acid at 60° to be converted easily, in a good yield, into a syrupy 5-



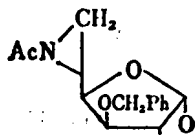
# I



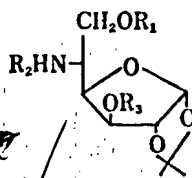
## III


$$\text{III: } R_1 = R_2 = \text{Ac}, R_3 = \text{CH}_2\text{Ph}$$

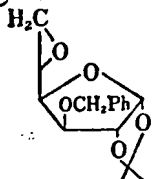
IV :  $R_1 = H$ ,  $R_2 = Ac$ ,  $R_3 = CH_2Ph$

$$V: R_1 = R_2 = R_3 \neq H$$


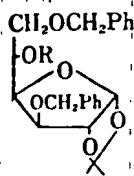
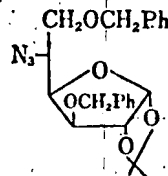
**CF:**


$$VII: R_1=R_2=Ac, R_3=CH_2Ph$$

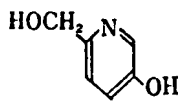
VIII:  $R_1=R_2=R_3=H$



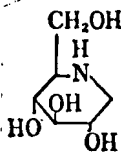
12


$$X:R=H$$
XI:  $R = Ts$ 

XII



XIII



XIV

- 1) H. Saeki, T. Iwashige, and E. Ohki, *Chem. Pharm. Bull.* (Tokyo), **16**, 188 (1968).
- 2) a) N. Ishida, K. Kumagai, T. Niida, T. Tsuruoka, and H. Yumoto, *J. Antibiotics* (Tokyo), Ser. A, **20** (2), 66 (1967); b) S. Inouye, T. Tsuruoka, and T. Niida, *J. Antibiotics* (Tokyo), Ser. A, **19** (6), 288 (1966).

acetamido-6-acetoxy  
give 5-acetamido-3-  
144°,  $[\alpha]_D^{25}$  -7.4° ( $c$ =  
Found: C, 61.33; H,  
IV, followed by sap  
1,2-O-isopropylidene  
(*Anal.* Calcd. for  $C_{17}H_{25}NO_6$   
which was identified  
that the attack of a  
terminal position.

Similar to the  $\alpha$ -O-isopropylidene- $\alpha$ -D-6-acetoxy compound  $C_{20}H_{27}O_7N$ : C, 61.05; H, 6.99; N, 6.39, of VII over palladium gave 5-amino-5-deoxy ( $c=1.1$ ,  $H_2O$ ) (Lit. m.p. 88–89°,  $[a]_D^{25} -13.7^\circ$ ,  $H_{21}O_{10}S$ : C, 64.97; H, 6.40; N, 9.88. Found: C, 64.97; H, 6.40; N, 9.88. Fused to charcoal at 70–80°).

VIII was quite preliminary treatment 289—290 m $\mu$  (in dil. of a pyridine derivat by acid treatment of O- and N-functions treatment of VIII w a syrupy trifluoroac Hydrolysis of the tr followed by removal afforded, in a good y 105° (decomp., soften H, 7.31; N, 7.82. F of Dowex 1 $\times$ 2(OH<sup>-</sup>) of infrared spectra :

- 3) a) R.E. Gramera, I Paulsen and K. Tod
- 4) Professor Whistler (incorrectly. Our sa test, and infrared sp
- 5) R.L. Whistler and R
- 6) A.S. Meyer and T. l



-011: 547.457/1.07

thesized a mono-  
of *Streptomyces*  
*arcina lutea* and

dofuranose<sup>1)</sup> (II)  
into a syrupy 5-

$\text{H}_2\text{Ph}$   
 $\text{=CH}_2\text{Ph}$

acetamido-6-acetoxy compound (III), which was deacetylated with sodium methoxide to give 5-acetamido-3-O-benzyl-5-deoxy-1,2-O-isopropylidene- $\beta$ -L-idofuranose (IV), mp 143–144°,  $[\alpha]_D^{25}$   $-7.4^\circ$  ( $c=5.3$ ,  $\text{CHCl}_3$ ) (Anal. Calcd. for  $\text{C}_{18}\text{H}_{23}\text{O}_6\text{N}$ : C, 61.52; H, 7.17; N, 3.99. Found: C, 61.33; H, 7.23; N, 4.05). The structure of IV was confirmed by hydrogenation of IV, followed by saponification with barium hydroxide solution to give 5-amino-5-deoxy-1,2-O-isopropylidene- $\beta$ -L-idofuranose (V), mp 185° (reported mp 178°<sup>3a)</sup>; mp 176–179°<sup>3b)</sup>). (Anal. Calcd. for  $\text{C}_9\text{H}_{17}\text{O}_5\text{N}$ : C, 49.30; H, 7.82; N, 6.39. Found: C, 49.00; H, 7.89; N, 6.47.), which was identified with the sample synthesized by the known method.<sup>3a)</sup> This fact indicated that the attack of an acetate ion on the 5,6-epimino ring of II was uniquely effected at the terminal position.

Similar to the case of II, treatment of 5,6-acetylepimino-3-O-benzyl-5,6-dideoxy-1,2-O-isopropylidene- $\alpha$ -D-glucofuranose<sup>1)</sup> (VI) with acetic acid afforded a crystalline 5-acetamido-6-acetoxy compound (VII), mp 123.5–124.5°,  $[\alpha]_D^{25}$   $-25.9^\circ$  ( $c=6.6$ ,  $\text{CHCl}_3$ ) (Anal. Calcd. for  $\text{C}_{20}\text{H}_{27}\text{O}_7\text{N}$ : C, 61.05; H, 6.92; N, 3.56. Found: C, 61.01; H, 6.98; N, 3.75.). Hydrogenation of VII over palladium-charcoal, followed by saponification with barium hydroxide solution, gave 5-amino-5-deoxy-1,2-O-isopropylidene- $\alpha$ -D-glucose (VIII), mp 125–126°,  $[\alpha]_D^{25}$   $-17.0^\circ$  ( $c=1.1$ ,  $\text{H}_2\text{O}$ ) (Lit. mp 86°,<sup>4)</sup>  $[\alpha]_D^{25}$   $-12.2^\circ$ <sup>5)</sup>) (Anal. Calcd. for  $\text{C}_9\text{H}_{17}\text{O}_5\text{N}$ : C, 49.30; H, 7.82; N, 6.39. Found: C, 49.64; H, 7.79; N, 6.39.). The structure of VIII was also confirmed by identification with the sample synthesized by a modification of Whistler's procedure;<sup>5)</sup> treatment of 5,6-anhydro-3-O-benzyl-1,2-O-isopropylidene- $\beta$ -L-idofuranose<sup>6)</sup> (IX) with sodium benzyolate, followed by tosylation of the resulting 3,6-dibenzyloxy compound (X), mp 74.5° (reported mp 89–90°<sup>6)</sup>) (Anal. Calcd. for  $\text{C}_{23}\text{H}_{28}\text{O}_6 \cdot \frac{1}{2}\text{H}_2\text{O}$ : C, 67.46; H, 7.15. Found: C, 67.57; H, 6.99.), afforded a crystalline 3,6-dibenzyloxy-5-tosyloxy compound (XI), mp 88–89°,  $[\alpha]_D^{25}$   $-13.7^\circ$  ( $c=2.6$ ,  $\text{CHCl}_3$ ) (Lit. mp 75–76°,  $[\alpha]_D^{25}$   $-15.3^\circ$ <sup>6)</sup>) (Anal. Calcd. for  $\text{C}_{30}\text{H}_{34}\text{O}_8\text{S}$ : C, 64.97; H, 6.18; S, 5.78. Found: C, 65.02; H, 6.21; S, 5.78.). XI was further treated with sodium azide, giving 5-azido-3,6-di-O-benzyl-5-deoxy-1,2-O-isopropylidene- $\alpha$ -D-glucose (XII), mp 69°,  $[\alpha]_D^{25}$   $-36.9^\circ$  ( $c=2.9$ ,  $\text{CHCl}_3$ ) (Anal. Calcd. for  $\text{C}_{23}\text{H}_{27}\text{O}_5\text{N}_3$ : C, 64.92; H, 6.40; N, 9.88. Found: C, 64.83; H, 6.50; N, 9.97.), which hydrogenated over palladium-charcoal at 70–80° and 70 kg/cm<sup>2</sup> to give VIII.

VIII was quite unstable to acids; it was found that a reaction product obtained by a preliminary treatment of VIII with acids exhibited absorption maxima at 225–226 m $\mu$ , 289–290 m $\mu$  (in dil. HCl) and 245 m $\mu$ , 302–303 m $\mu$  (in dil. NaOH), suggesting the presence of a pyridine derivative (XIII) or the likes, which would be the same compound obtained by acid treatment of nojirimycin.<sup>2b)</sup> However, hydrolysis of VIII was successful by protecting O- and N-functions with easily-removable trifluoroacetyl group before acid treatment; treatment of VIII with trifluoroacetic anhydride in acetonitrile without bases to easily gave a syrupy trifluoroacetate which showed no hydroxyl absorption in its infrared spectrum. Hydrolysis of the trifluoroacetate with 0.1 N diluted hydrochloric acid at 70–80° for 1 hr, followed by removal of the protective group by basification to pH 7–8 with Dowex 1 $\times$ 4 (OH<sup>-</sup>), afforded, in a good yield, an amorphous D-glucopiperidinose (I). The analytical sample, mp 105° (decomp., softening at 95°),  $[\alpha]_D^{25}$   $+63^\circ$  ( $c=1.2$ ,  $\text{H}_2\text{O}$ ) (Anal. Calcd. for  $\text{C}_6\text{H}_{13}\text{O}_3\text{N}$ : C, 40.22; H, 7.31; N, 7.82. Found: C, 40.82; H, 6.97; N, 7.86.), purified by passing through a column of Dowex 1 $\times$ 2 (OH<sup>-</sup>), was identified with the authentic sample of nojirimycin by comparison of infrared spectra and thin-layer chromatography. Moreover, hydrogenation of I over

3) a) R.E. Gramera, R.M. Bruce, S. Hirase, and R.L. Whistler, *J. Org. Chem.*, **28**, 1401 (1963); b) H. Paulsen and K. Todt, *Chem. Ber.*, **99**, 3450 (1966).

4) Professor Whistler (private communication) said that the melting point of VIII recorded<sup>3)</sup> was listed incorrectly. Our sample of VIII was identified with the sample sent from him by mixed melting point test, and infrared spectrometry.

5) R.L. Whistler and R.E. Gramera, *J. Org. Chem.*, **29**, 2609 (1964).

6) A.S. Meyer and T. Reichstein, *Helv. Chim. Acta*, **29**, 152 (1946).



platinum gave a deoxy-compound (XIV), mp 195°,  $[\alpha]_D^{25} +43.1^\circ$  ( $c=1.4$ ,  $H_2O$ ) (reported mp 195°,  $[\alpha]_D^{25} +47^\circ$ ), which was also identical with the sample derived from the natural antibiotic by comparison of infrared spectra and mixed melting point test. The synthesized nojirimycin (I) also showed the same activity against *Sarcina lutea* and *Xanthomonas oryzae*.

**Acknowledgement** We wish to express our appreciation to Dr. T. Ito<sup>7)</sup> of Central Research Laboratories, Meiji Seika Kaisha, Ltd., for a generous gift of nojirimycin and its derivative, and to Professor R.L. Whistler of Purdue University, Indiana, U.S.A. for sending us the sample of VIII. We are also indebted to Dr. G. Sunagawa, Director, and Dr. I. Iwai, Assistant Director of this Laboratories for their encouragement, and to Mr. Y. Shimada for his technical assistance.

Central Research Laboratories,  
Sankyo Co., Ltd.  
Hiromachi, Shinagawa-ku, Tokyo

HIROMICHI SAEKI  
Eiji Ojiki

Received February 23, 1968

7) According to Dr. Ito's personal communication, his group has accomplished independent synthesis of nojirimycin from D-glucose (S. Inouye, T. Tsuruoka, T. Ito, and T. Niida, *Tetrahedron* 24, 2125 (1968)).

## TWO NEW

### FRUCTOSE-6-PH

Sodium S  
Grade 1 Approx  
100 mg—\$  
500 mg—\$  
1 g — 1  
5 g — 4

Free of Fructose  
and contains less than  
Glucose-6-Phosphate

As many of you  
pounds with per  
many years of u  
nique that really  
be shipped pro

WE

THE FIR

### (-) CITRAMA

Sigma Gr  
Sodium S  
25 mg—\$  
100 mg—\$  
500 mg—\$  
1 g —

### NOW PRODUCE

### GUANO

Grade 1 in  
100 mg—\$

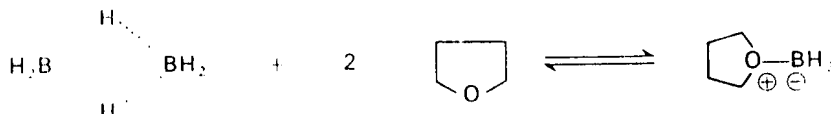
### GUANO

Grade 1 S  
100 mg—\$5

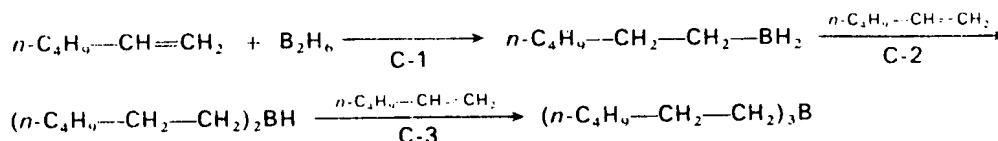
PLEASE  
THE SIGN  
THERE IS  
IF SHIPP

SIGMA LO

hydrofuran solution of diborane, probably present in equilibrium with the illustrated tetrahydrofuran-borane complex, is free from inorganic contaminants and may be standardized by reaction of an aliquot of the solution with excess acetone followed by addition of a known excess of standard aqueous alkali to saponify the borate ester present. The aqueous solution is then treated with mannitol and titrated with standard acid.



Although diborane is capable of reducing a variety of functional groups (Table 2-2),<sup>39</sup> the most useful synthetic applications of the reagent have involved addition to carbon-carbon multiple bonds, called hydroboration. The rate of addition of diborane to carbon-carbon double bonds decreases as the number of alkyl substituents about the double bond increases.<sup>40</sup> With mono- and disubstituted



olefins, all three hydrogen atoms of the borane are used in the reduction.<sup>41</sup> Each successive addition step is slower than the preceding step (i.e., C-1 > C-2 > C-3), a result that has been attributed to the increasing steric bulk of the partially alkylated boron reagent. In agreement with this idea, trisubstituted olefins normally

(39) (a) H. C. Brown and B. C. Subba Rao, *J. Am. Soc.*, **82**, 681 (1960); (b) H. C. Brown and W. Korytnyk, *ibid.*, **82**, 3866 (1960); (c) H. C. Brown and D. B. Bigley, *ibid.*, **83**, 486 (1961); (d) carboxyl and cyano functions have been selectively reduced in the presence of ketone functions with diborane; see B. C. Subba Rao and G. P. Thakar, *Current Sci. (India)*, **32**, 404 (1963); *C.A.*, **60**, 438 (1964); (e) for the reduction of enamines with sodium borohydride and acetic acid (a procedure that involves reaction of the iminium salt with diborane), see J. A. Marshall and W. S. Johnson, *J. Org. Chem.*, **28**, 421 (1963); (f) the pyridine-borane complex in acetic acid solution is also a useful reducing agent for ketones; see K. V. Yorka, W. L. Truett, and W. S. Johnson, *ibid.*, **27**, 4580 (1962).

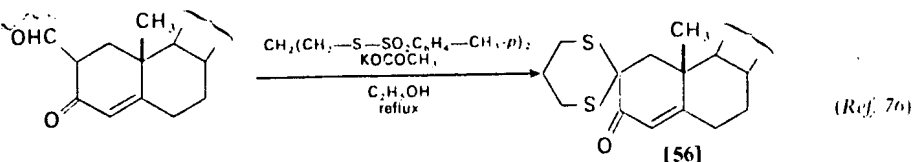
(40) H. C. Brown and G. Zweifel, *J. Am. Chem. Soc.*, **82**, 3222, 3223, 4708 (1960).

(41) (a) H. C. Brown and B. C. Subba Rao, *ibid.*, **81**, 6423, 6428 (1959); (b) H. C. Brown and A. W. Moerikofer, *ibid.*, **84**, 1478 (1962); **85**, 2063 (1963).

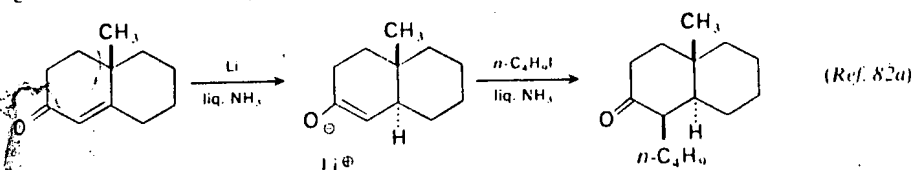
[55]

(94%)

$\alpha$ -formylketone with 1,3-propanedithiol di-*p*-toluenesulfonate in alcoholic potassium acetate yields a keto dithioketal (e.g., [56]), in which the dithioketal function serves to block a methylene group during alkylation<sup>76</sup>; it is removed by hydrogenolysis over Raney nickel (see Chapter 1). The previously discussed monoalkylation of the dianion derived from an  $\alpha$ -formylketone can also be considered an example of the use of a blocking group.



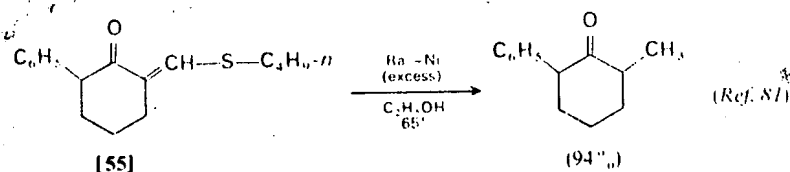
A method for selectively generating a specific enolate anion that has received limited application thus far consists of reduction of an  $\alpha,\beta$ -unsaturated ketone or of an  $\alpha$ -halo- or  $\alpha$ -acetoxyketone with a metal such as lithium, sodium, or calcium in liquid ammonia<sup>82</sup> (see Chapter 3). As illustrated in the equation below, enolate anions generated in this way can be alkylated before equilibration occurs.



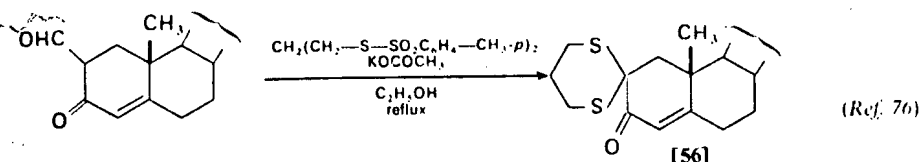
An indirect procedure for the selective alkylation (or acylation; see Chapter 9) of an aldehyde or ketone involves the initial reaction of the carbonyl compound with a secondary amine to form an intermediate enamine.<sup>83</sup> Typical enamine preparations are outlined in the accompanying equations. It will be noted that the less

(82) (a) G. Stork, P. Rosen, and N. L. Goldman, *J. Am. Chem. Soc.*, **83**, 2965 (1961); (b) R. E. Schaub and M. J. Weiss, *Chem. Ind. (London)*, **1961**, 2003; (c) M. J. Weiss, R. E. Schaub, J. F. Poletto, G. R. Allen, Jr., and C. J. Coscia, *ibid.*, **1963**, 118; (d) M. J. Weiss and co-workers, *Tetrahedron*, **20**, 357 (1964).

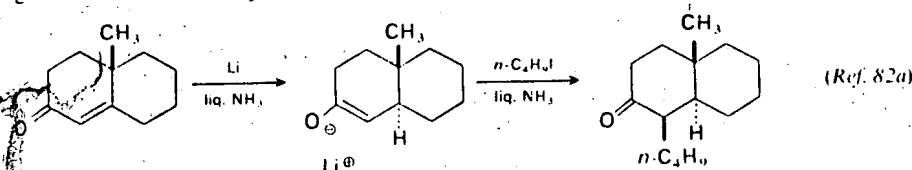
(83) (a) G. Stork, A. Brizzolara, H. Landesman, J. Szmuszkowicz, and R. Terrell, *J. Am. Chem. Soc.*, **85**, 207 (1963); (b) J. Szmuszkowicz in R. A. Raphael, E. C.



$\alpha$ -formylketone with 1,3-propanedithiol di-*p*-toluenesulfonate in alcoholic potassium acetate yields a keto dithioketal (e.g., [56]), in which the dithioketal function serves to block a methylene group during alkylation<sup>76</sup>; it is removed by hydrogenolysis over Raney nickel (see Chapter 1). The previously discussed monoalkylation of the dianion derived from an  $\alpha$ -formylketone can also be considered an example of the use of a blocking group.



A method for selectively generating a specific enolate anion that has received limited application thus far consists of reduction of an  $\alpha,\beta$ -unsaturated ketone or of an  $\alpha$ -halo- or  $\alpha$ -acetoxyketone with a metal such as lithium, sodium, or calcium in liquid ammonia<sup>82</sup> (see Chapter 3). As illustrated in the equation below, enolate anions generated in this way can be alkylated before equilibration occurs.

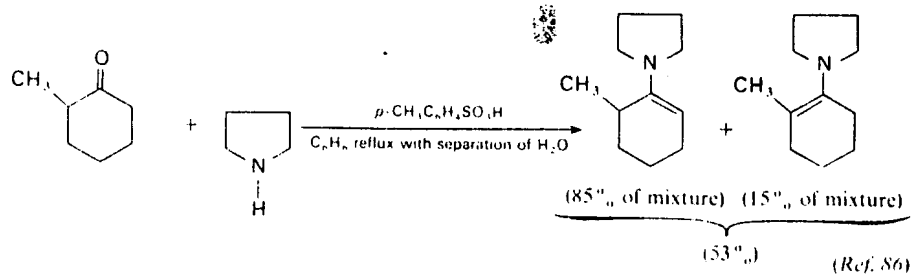
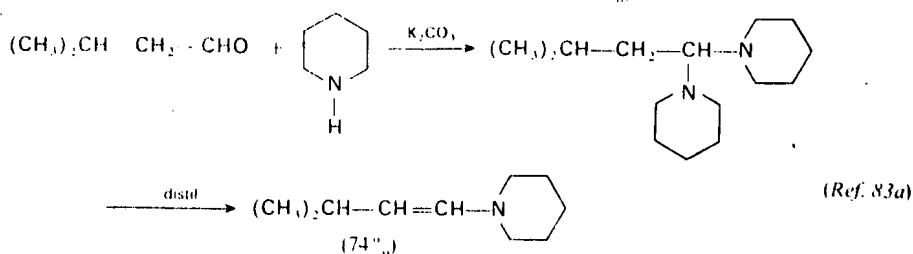
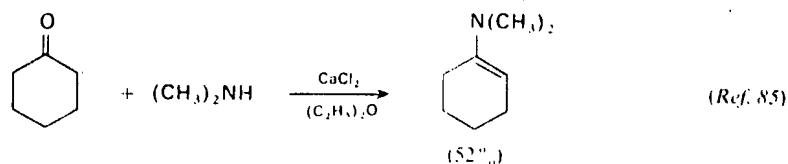
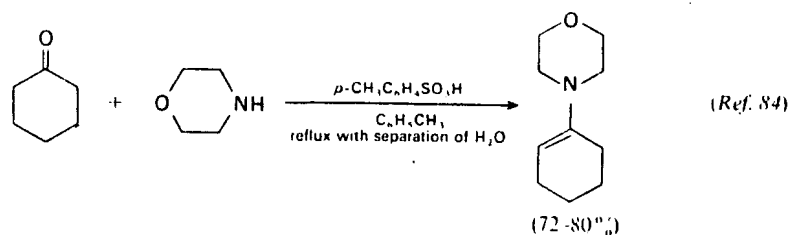


An indirect procedure for the selective alkylation (or acylation; see Chapter 9) of an aldehyde or ketone involves the initial reaction of the carbonyl compound with a secondary amine to form an intermediate enamine.<sup>83</sup> Typical enamine preparations are outlined in the accompanying equations. It will be noted that the less

(82) (a) G. Stork, P. Rosen, and N. L. Goldman, *J. Am. Chem. Soc.*, **83**, 2965 (1961); (b) R. E. Schaub and M. J. Weiss, *Chem. Ind. (London)*, **1961**, 2003; (c) M. J. Weiss, R. E. Schaub, J. F. Poletto, G. R. Allen, Jr., and C. J. Cosecia, *ibid.*, **1963**, 118; (d) M. J. Weiss and co-workers, *Tetrahedron*, **20**, 357 (1964).

(83) (a) G. Stork, A. Brizzolara, H. Landesman, J. Szmuszkowicz, and R. Terrell, *J. Am. Chem. Soc.*, **85**, 207 (1963); (b) J. Szmuszkowicz in R. A. Raphael, E. C.

highly substituted enamine is usually the predominant product unless the enamine function can be stabilized by conjugation with an adjacent functional group. The electronic distribution in enamines, as exemplified by the resonance structures [57] and



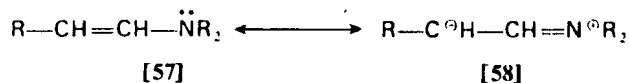
Taylor, and H. Wynberg (ed.), *Advances in Organic Chemistry: Methods and Results*, Vol. 4, Wiley-Interscience, New York, 1963, pp. 1-113.

(84) S. Hünig, E. Lücke, and W. Brenninger, *Org. Syn.*, **41**, 65 (1961).

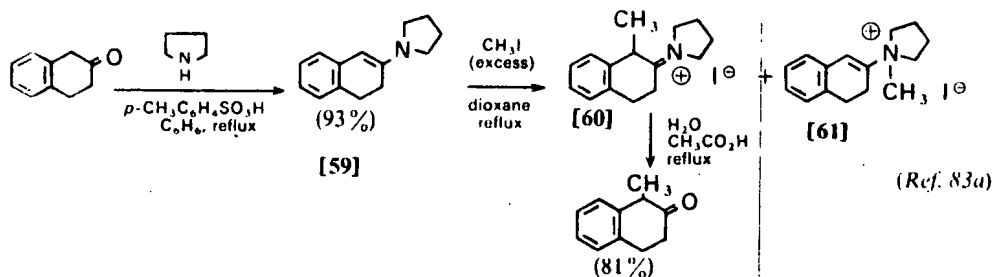
(85) E. P. Blanchard, Jr., *J. Org. Chem.*, **28**, 1397 (1963).

(86) (a) M. E. Kuchne, *J. Am. Chem. Soc.*, **81**, 5400 (1959); (b) H. O. House and M. Schellenbaum, *J. Org. Chem.*, **28**, 34 (1963).

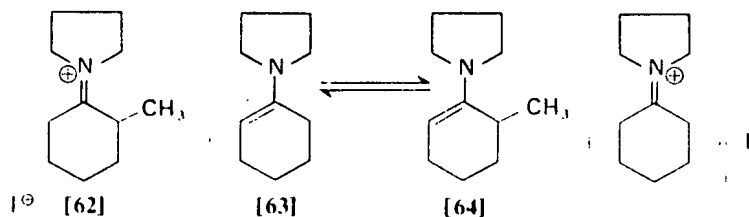
[58], is such that the beta carbon atom bears an appreciable negative charge and may serve as a nucleophile. When enamines (e.g., [59])



are treated with alkylating agents, reaction paths leading to C-alkylation or N-alkylation are possible. Subsequent hydrolysis of the C-alkylated iminium salt [60] yields an alkylated ketone, whereas the N-alkylated product [61] is usually water soluble, and relatively inert to hydrolysis. This alkylation procedure does not



preclude the possibility of alkylation since the iminium salt (e.g., [62]) can react with the original enamine (e.g., [63]) to form a new enamine (e.g., [64]), which can react with the alkylating agent. In

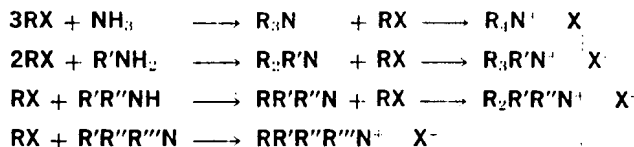


such cases, hydrolysis of the reaction mixture yields, as neutral products, a mixture of the starting ketone (from the salt of the starting enamine), the monoalkylated product, and the dialkylated product(s). Frequently, the alkylation of enamines with simple alkylating agents is not a good preparative method because the major reaction is N-alkylation rather than C-alkylation.<sup>83</sup> However, good yields of C-alkylated products have been obtained with such very reactive alkylating agents as allyl halides, benzyl halides, or  $\alpha$ -halocarbonyl compounds. The successful use of these reagents (e.g., [67]) may be attributable to the ability of initially formed

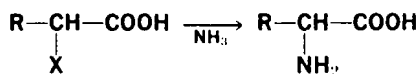
936, 280 APPLICATION FILE COPY  
Nitrogen Nucleophiles

A. Attack by  $\text{NH}_3$ ,  $\text{NHR}$ , or  $\text{NR}_2$  at an alkyl carbon

0-44 Alkylation of amines



The reaction between alkyl halides and ammonia or primary amines is not usually a feasible method for the preparation of primary or secondary amines, since they are stronger bases than ammonia and preferentially attack the substrate. However, the reaction is very useful for the preparation of tertiary amines<sup>205</sup> and quaternary ammonium salts. If ammonia is the nucleophile, the three or four alkyl groups on the nitrogen of the product must be identical. If a primary, secondary, or tertiary amine is used, then different alkyl groups may be placed on the same nitrogen atom. The conversion of tertiary amines to quaternary salts is called the *Menschutkin reaction*. It is sometimes possible to use this method for the preparation of a primary amine, by the use of a large excess of ammonia, or of a secondary amine, by the use of a large excess of primary amine. However, the limitations of this approach may be seen in the reaction of a saturated solution of ammonia in 90% ethanol with ethyl bromide in a 16:1 molar ratio, under which conditions the yield of primary amine was 34.2% (at a 1:1 ratio the yield was 11.3%).<sup>206</sup> In some cases, where inductive or other effects cause the primary amine to be a weaker base than ammonia, it can easily be prepared in good yield. An important example is the conversion of  $\alpha$ -halo acids to  $\alpha$ -amino acids:



Primary amines may be prepared from alkyl halides by method 0-45 or, more commonly, by the Gabriel synthesis (0-57).

The conjugate bases of ammonia and of primary and secondary amines ( $\text{NH}_2^-$ ,  $\text{RNH}^-$ ,  $\text{R}_2\text{N}^-$ ) are sometimes used as nucleophiles. However, they offer no advantages over ammonia or the amines themselves, since the latter are basic enough. This is in contrast to the analogous methods 0-1, 0-16, 0-36, and 0-37. Diaryl and triaryl amines are very poor nucleophiles, although the reaction has been carried out with diarylamines.<sup>207</sup> Sulfates or sulfonates may be used instead of halides. As usual, tertiary substrates do

<sup>205</sup>For a review of this reaction with particular emphasis on the preparation of tertiary amines, see Spialter and Pappalardo, "The Acyclic Aliphatic Tertiary Amines," pp. 14-29, The Macmillan Company, New York, 1965.

<sup>206</sup>Werner, *J. Chem. Soc.* **113**, 899 (1918).

<sup>207</sup>Patai and Weiss, *J. Chem. Soc.* **1959**, 1035.

from trifluoroacetates,  $^+CF_3$ . The pyrylium ion is characteristic for the acetates and trifluoroacetates. A typical reaction of acetates is the decomposition of two groups with the elimination of ketene and  $Ac_2O$ . Trifluoroacetates do not behave thus, but eliminate  $CF_3CO_2H$ . The decomposition of 1,6-acetamino sugars follows a different route, leading to a stable end product which retains the intact ring.

D. H. Hutson

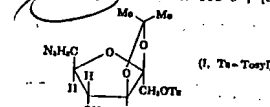
**65779t** Thin-layer chromatography of carbohydrates. A. Pastuszyn and H. Michl. *Mitt. Versuchsanst. Gärungsgewerbe Wien* 20(1-2), 1-4 (1966) (Ger.). To separate and detect 0.04-0.08  $\mu$ mole amts. of carbohydrates by thin-layer chromatography, develop the mixt. on Silica Gel G twice in the same direction with  $BuOH:EtOH:H_2O$  (5:1:4), drying between runs, then once transversely with  $BuOH:HOAc:EtOH:H_2O$  (9:6:3:1). Let the solvent front move 14 cm. each time. Spray 1st with 1:2  $2N$   $PhNH_2$  in  $BuOH:2N$   $H_2PO_4$ , heat 10 min. at  $110^\circ$  to detect almost all compds. by uv fluorescence. Spray again with 10:1 0.4% orcinol in  $EtOH$ -conc.  $H_2PO_4$ ; heat as before to detect ketoses, sucrose, and raffinose. Spray a 3rd time with 10:1 0.4% naphthoresorcinol in  $EtOH$ -conc.  $H_2PO_4$ ; heat as before to detect aldopentoses, aldohexoses, ketoses, and other sugars as yellow-brown spots. Development time totals 7.5 hrs.  $C_{12-14}$  compds. cannot be sep'd. because of the reaction with silica gel. Oligosaccharides such as maltotriose and maltohexose are sep'd. better with  $BuOH$ -iso- $PrOH:H_2O$  mixts. Historical diagrams are shown for the sep'n. and detection of arabinose, 2-deoxy- $\alpha$ -arabino-hexose, 2-deoxy-erythro-pentose, fructose, fucose, galactose, 2-amino-2-deoxyglucose, glucose, lactose, lyxose, maltose, mannose, 2-acetamido-2-deoxyglucose, raffinose, rhamnose, ribose, sucrose, sorbose, tagatose, trehalose, xylose, glucuronic acid, maltotetraose, melibiose, cellobiose, "dendroketo" (I), digitoxose, 5-hydroxymethylfurfural (II), galacturonic acid, maltotriose, and melizitose in synthetic mixts., in wine, and in the condensation product of  $HCHO$  (III). Prep. I by treating 1 g.  $CO(CH_3OH)_2$  with 20 ml. of 0.5N  $NaOH$  at room temp. for 4 hrs. Neutralize with conc.  $HCl$ , steam-distill, and ext. the residue with 80%  $EtOH$ . Conc. the ext., then sep. I by paper chromatography. Prep. II by heating 10 g. sucrose with 30 ml. of aq. 0.3%  $COOH$  in a closed vessel at  $145^\circ$  for 15 min., then at  $125^\circ$  for 2 1/2 hrs. Cool, neutralize with  $CaCO_3$ , and ppt. impurities with 0.5 g.  $Ph(OAc)_3$ . Ext. with  $EtOAc$ , dry over anhyd.  $MgSO_4$ , distill off the solvent, and crystallize II (m.p.  $31^\circ$ ) from the brown sirup. Prep. III by heating 0.1M  $HCHO$  in aq. 0.04N  $LiOH$  and 0.001N  $Ca(OH)_2$  for 14 min. Cool quickly, desalt with Dowex 50, steam distill, dissolve in  $MeOH$ , and steam distill repeatedly to remove the residual  $HCHO$ .

J. Bernstich

**65780m** Methylation of carbohydrates bearing base-labile substituents, with diazomethane-boron trifluoride etherate. I. O. Mastroratti, S. M. Pignatti, J. O. Deferrari, and E. G. Gros (Univ. Buenos Aires). *Carbohydr. Res.* 3(2), 177-83 (1966) (Eng.). Various tetra-O-acetyl-D-hexopyranoses undergo acetyl migration on methylation with Purdie's reagents; thus, 1,2,3,4-tetra-O-acetyl- $\beta$ -D-glucopyranose, 1,2,3,6-tetra-O-acetyl- $\beta$ -D-glucopyranose, and 1,2,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranose and its anomers are converted into methyl 2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside when methylated with  $MeI$  and  $Ag_2O$ . When the compd. is treated with  $CH_3N_2$  and  $BF_3 \cdot Et_2O$ , the methylated product is produced in a high yield, with the  $Me$  ether group in the expected position. Under the same conditions, 1,3,4,6-tetra-O-acetyl- $\alpha$ -D-galactopyranose (I) is converted into the 2-Me ether without the acetyl migration observed when I was methylated with Purdie's reagents. The tetra-O-acetyl-O-methyl-D-hexopyranoses were transformed by deacetylation into the following mono-O-methylhexoses: 2-O-methyl- $\beta$ -D-glucose, 4-O-methyl-D-glucose, 6-O-methyl- $\alpha$ -D-glucose, and 2-O-methyl- $\beta$ -D-galactose. 10 references. RCYM

**65781n** Synthesis of hydropiperidines from carbohydrate precursors. Stephen Ilanessian (Parke, Davis & Co., Ann Arbor, Mich.). *Chem. Ind. (London)* 1966(51), 2126-7 (Eng.). Redn. of 5-azido-5-deoxy-pentoses in  $MeOH$  in the presence of 6 or 20%  $Pd/C$  at room temp. and atm. pressure for 1-2 hrs. afforded the corresponding 3,4,5-trihydroxypiperidines, characterized as the N-acetyl derivatives, in a very good yield, e.g. N-acetyl-3,4,5-trihydroxypiperidine, m.  $193-4^\circ$  (c 3.10  $H_2O$ ) in quant. yield. Reductive rearrangement of 6-azido-2,3-O-benzylidene-5-deoxy- $\beta$ -D-ribofuranose afforded after N-acetylation, N-acetyl-3,4-O-benzylidene-D-ribo-3,4,5-trihydroxypiperidine, m.  $154-6^\circ$ ,  $[\alpha]_D^{25} -42.7^\circ$  (c 1.53  $MeOH$ ). 6-Azido-6-deoxy-D-glucose, m.  $128-30^\circ$ , or the D-galacto analog, m.  $145-5^\circ$ , gave after acetylation the corresponding 6-acetamido-6-deoxyhexoses rather than piperidine products with N in the ring. Redn. of 6-azido-5-deoxy-D-glucosylactone, m.  $210-12^\circ$  (decolorn.). Reaction of 2,3-O-isopropylidene-1,6-di-O-p-tolylmethyl- $\beta$ -fructofuranose with 1 molar equiv. of  $Na_2N_2$  in  $HCO_2Me$  at  $100^\circ$  for 18 hrs. gave 6-azido-6-deoxy-2,3-O-isopropylidene-1-O-p-tolylmethyl- $\beta$ -fructofuranose (I), m.  $76-8^\circ$ . Reduction of the p-tolylmethyl ether function in I by a benzoate subsequent deacetylation and hydrolysis afforded 6-azido-6-deoxy-D-glucose (II). Reductive rearrangement of II

gave a 2-hydroxymethyl-2,4,5-trihydroxypiperidine,  $[\alpha]_D^{25} -11.5^\circ$  (c 3  $MeOH$ ). N-acetyl deriv. m.  $198-9^\circ$ ,  $[\alpha]_D^{25} -11^\circ$  (c 0.4  $MeOH$ ). Reductive rearrangement of 5-azido-5,6-dideoxy-L-talose and subsequent acetylation gave N-acetyl-5-methyl-L-talo-3,4,5-trihydroxypiperidine,  $[\alpha]_D^{25} 11.4^\circ$  (c 1.55  $MeOH$ ).

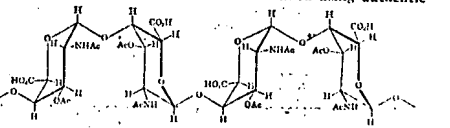


**65782p** Sequence studies on *Diplococcus pneumoniae* type II polysaccharide. S. A. Barker, P. J. Somers, and M. Stacey (Univ. Birmingham, Eng.). *Carbohydr. Res.* 3(3), 261-70 (1967) (Eng.). Assay of  $HCO_2H$  released by periodate oxidn. of *D. pneumoniae* Type II polysaccharide (SII), during liberation of an L-rhamnose-contg. trisaccharide from SII by  $\alpha$ -L-rhamnosidase, demonstrated an  $\alpha$ -(1  $\rightarrow$  4) linkage between this trisaccharide and the remainder of the SII mol. Carboxyl-reduced SII,  $[\alpha]_D^{25} 42^\circ$ , contg. 49.7% D-glucose and 49.8% L-rhamnose was prep'd. by redu. of the 2-hydroxyethyl ester in two stages. A comparison of the cleaved products of the action of specific, induced  $\alpha$ - and  $\beta$ -D-glucosidases on SII and carboxyl-reduced SII demonstrated the presence of O- $\beta$ -(D-glucopyranosyluronic acid)-(1  $\rightarrow$  4)-O- $\alpha$ -D-glucopyranosyl-(1  $\rightarrow$  4) residues in SII and O- $\beta$ -D-glucopyranosyl-(1  $\rightarrow$  4)-O- $\alpha$ -D-glucopyranosyl-(1  $\rightarrow$  4) residues in the reduced polysaccharide. A possible repeating unit for SII is discussed. RCYM

**65783q** Influence of the hydrogen ion concentration on the specificity of the TTC reaction. Klaus Lorenz and Hans Hoffmeister (Univ. Hamburg, Ger.). *Mikrochim. Acta* 105(6), 1002-7 (Ger.). Triphenyltetrazolium chloride (TTC), is widely used as a dehydrogenation reagent for org. compds. The  $H_2O$ -sol. TTC salt is converted during this reaction into the corresponding formazan, a red insol. compd. The spontaneous formation of formazan occurs only in alk. media and the number of compds. responding to dehydrogenation increases with increasing pH of the environment. The threshold pH of 12.45, above which TTC remains inactive, could be lowered to 12.0 by addn. of  $MeOH$  or  $tert$ - $BuOH$ . This shift reduces the no. of reagent partners, increases the specificity of the dehydrogenation, and allows one to follow quant. the progress of reaction. Data on D-glucose, D-glucose 1-phosphate, sucrose, cellulose powder, coll. starch, glycogen, 2-amino-2-deoxy-D-glucose, hydrochloride, chondroitin sulfate, hyaluronic acid, heparin, Na lactate, Na K tartrate, Na pyruvate,  $\alpha$ -oxoglutaric acid, cysteine hydrochloride, creatinine, urea, and glycine are tabulated. G. W. Scherf

**65784r** Preparation and characterization of 2-O-(4-O-methyl- $\alpha$ -D-glucopyranosyl)-xylopyranose. Nirmolendu Roy and T. E. Timell (State Univ., Coll. of Forestry, Syracuse, N.Y.). *Carbohydr. Res.* 3(2), 249-7 (1966-1967) (Eng.). 2-O-(4-O-Methyl- $\alpha$ -D-glucopyranosyl)-xylopyranose, prep'd. by partial acid hydrolysis of a (4-O-methylglucuronoxylan from aspen wood, was acetylated with  $Ac_2O$ - $NaOAc$ . The product was reduced with  $B_2H_6$  in tetrahydrofuran and saponified with  $NaOMe$  to give 67% title compd., m.  $95-105^\circ$ ,  $[\alpha]_D^{25} 114^\circ$  ( $H_2O$ ). Derek Horton

**65785a** Elucidation of the structure of the Vi-antigen isolated from *Escherichia coli* 5396/38. K. Heyns and G. Kicsling (Univ. Hamburg, Ger.). *Carbohydr. Res.* 3(3), 340-53 (1967) (Ger.). The structure of the Vi-antigen isolated from *E. coli* 5396/38 was elucidated by applying (1) methylation followed by redn. and (2) redn. prior to methylation. Hydrolyzates of the Vi-antigen material treated in these two ways contained 2-amino-2-deoxy-3,6-di-O-methyl-D-galactopyranose (from 1) and 2-amino-2-deoxy-3,6-di-O-methyl-D-glucopyranose (from 2), resp. The occurrence of these 2 compds., as well as the absence of 2-amino-2-deoxy-4-O-methyl-D-galactopyranose and 2-amino-2-deoxy-4,6-di-O-methyl-D-galactopyranose, conclusively demonstrates 1  $\rightarrow$  4 glycosidic bonds in the polysaccharide. The products formed by hydrolysis were quant. det'd. and identified using authentic



control substances in the amino acid analysis of Moore, Spackman, and Stein by a special buffer. From these results a model of the structure of the Vi-antigen is presented. 20 references. RCYM



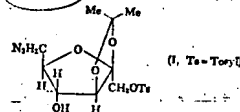
from trifluoroacetates,  $^4\text{CF}_3$ . The pyrylium ion is characteristic for the acetates and trifluoroacetates. A typical reaction of acetates is the decompn. of two groups with the elimination of ketene and  $\text{Ac}_2\text{O}$ . Trifluoroacetates do not behave thus, but eliminate  $\text{CF}_3\text{CO}_2\text{H}$ . The decompn. of 1,6-acetamino sugars follows a different route, leading to a stable end product which retains the intact ring.

**657791** Thin-layer chromatography of carbohydrates. A. Pastusyn and H. Michl. *Mitt. Versuchsst. Geringerwerke* 11 (1966) (Ger.). To sep. and detect 0.04-0.08  $\mu\text{mole}$  amts. of carbohydrates by thin-layer chromatog., develop the mixt. on Silica Gel G twice in the same direction with  $\text{BuOH}:\text{EtOH}:\text{H}_2\text{O}$  (5:1:4), drying between runs, then once transversely with  $\text{BuOH}:\text{HOAc}:\text{EtOH}:\text{H}_2\text{O}$  (9:6:3:1). Let the solvent front move 14 cm. each time. Spray 1st with 1:2  $\text{N}$   $\text{PhNH}_2$  in  $\text{BuOH}:\text{EtOH}$ ; heat 10 min. at  $110^\circ$  to detect almost all compds. by uv fluorescence. Spray again with 10:1 0.4% orcinol in  $\text{EtOH}:\text{conc. H}_2\text{SO}_4$ ; heat as before to detect ketoses, sucrose, and raffinose. Spray a 3rd time with 10:1 0.4% naphthoresorcinol in  $\text{EtOH}:\text{conc. H}_2\text{SO}_4$ ; heat as before to detect aldopentoses, aldohexoses, ketoses, and other sugars as yellow-brown spots. Development time totals 7.5 hrs.  $\text{C}_{12}$  compds. cannot be sep'd. because of the reaction with silica gel. Oligosaccharides such as maltotriose and maltohexose are sep'd. better with  $\text{BuOH}:\text{iso-PrOH}:\text{H}_2\text{O}$  mixts. Pictorial diagrams are shown for the sep'n. and detection of arabinose, 2-deoxy-arabino-hexose, 2-deoxy-xylo-pentose, fructose, fucose, galactose, 2-amino-2-deoxyglucose, glucose, lactose, lyxose, maltose, mannose, 2-acetamido-2-deoxyglucose, raffinose, rhamnose, ribose, sucrose, sorbose, tagatose, trehalose, xylose, glucuronic acid, maltotetraose, melibiose, cellobiose, "dendroketose" (I), digitoxose, 5-hydroxymethyl-furfural (II), galacturonic acid, maltotriose, and anelutose in synthetic mixts., in wine, and in the condensation-product of  $\text{HCHO}$  (III). Prep. I by treating 1 g.  $\text{CO}(\text{CH}_3)_2$  with 20 ml. of 0.5N  $\text{NaOH}$  at room temp. for 4 hrs. Neutralize with conc'd.  $\text{HCl}$ , steam-distill, and ext. the residue with 80%  $\text{EtOH}$ . Conc. the ext., then sep. I by paper chromatog. Prep. II by heating 10 g. sucrose with 30 ml. of aq. 0.3%  $(\text{COOH})_2$  in a closed vessel at  $145^\circ$  for 15 min., then at  $125^\circ$  for 24 hrs. Cool, neutralize with  $\text{CaCO}_3$ , and ppt. impurities with 0.5 g.  $\text{Pb}(\text{OAc})_2$ . Ext. with  $\text{EtOAc}$ , dry over anhyd.  $\text{MgSO}_4$ , distill off the solvent, and crystallize II (m.p.  $31^\circ$ ) from the brown sirup. Prep. III by heating 0.1M  $\text{HCHO}$  in aq. 0.04N  $\text{LiOH}$  and 0.001N  $\text{Ca}(\text{OH})_2$  for 14 min. Cool quickly, desalt with Dowex 50, steam distill, dissolve in  $\text{MeOH}$ , and steam distill repeatedly to remove the residual  $\text{HCHO}$ .

**65780m** Methylation of carbohydrates bearing base-labile substituents, with diazomethane-boron trifluoride etherate. I. O. Mastroratti, S. M. Menatti, J. O. DeFerrari, and E. C. Gross (Univ. Buenos Aires). *Carbohydr. Res.* 3 (2), 177-83 (1966) (Eng.). Various tetra-O-acetyl- $\beta$ -D-glucopyranosides undergo acetyl migration on methylation with Purdie's reagents; thus, 1,2,3,4-tetra-O-acetyl- $\beta$ -D-glucopyranose, 1,2,3,6-tetra-O-acetyl- $\beta$ -D-glucopyranose, and 1,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranose and its  $\alpha$ -anomer are converted into methyl 2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside when methylated with  $\text{MeI}$  and  $\text{Ag}_2\text{O}$ . When the compd. is treated with  $\text{CH}_3\text{N}_2$  and  $\text{BF}_3:\text{Et}_2\text{O}$ , the methyl product is produced in a high yield, with the  $\text{Me}$  ether group in the expected position. Under the same conditions, 1,2,4,6-tetra-O-acetyl- $\alpha$ -D-galactopyranose (I) is converted into the 2-Me ether without the acetyl migration observed when I was methylated with Purdie's reagents. The tetra-O-acetyl- $\alpha$ -D-methyl- $\beta$ -D-glucopyranosides were transformed by decetylation into the following mono-O-methylhexoses: 2-O-methyl- $\beta$ -D-glucose, 4-O-methyl- $\beta$ -D-glucose, 6-O-methyl- $\alpha$ -D-glucose, and 2-O-methyl- $\beta$ -D-galactose. 10 references. RCYM

**65781n** Synthesis of hydroperidines from carbohydrate precursors. Stephen Hanessian (Parke, Davis & Co., Ann Arbor, Mich.). *Chem. Ind. (London)* 1966 (51), 2126-7 (Eng.). Redn. of 5-azido-5-deoxyribose in  $\text{MeOH}$  in the presence of 6 or 20%  $\text{Pd/C}$  at room temp. and atm. pressure for 1-2 hrs. afforded the corresponding 3,4,5-trihydroxypiperidines, characterized as the  $\text{N}$ -acetyl derivs., in a very good yield, e.g.  $\text{N}$ -acetyl-3,4,5-trihydroxypiperidine, m.  $193-4^\circ$ ,  $[\alpha]_D^{25} 0^\circ$  (c 3.10  $\text{EtOH}$ ) in quant. yield. Reductive rearrangement of 5-azido-2,3,6-tri-O-benzylidene-5-deoxy- $\beta$ -D-ribofuranose afforded after  $\text{N}$ -acetylation,  $\text{N}$ -acetyl-3,4-O-benzylidene- $\beta$ -ribo-3,4,5-trihydroxypiperidine, m.  $154-5^\circ$ ,  $[\alpha]_D^{25} -42.7^\circ$  (c 1.53  $\text{MeOH}$ ). 6-Azido-6-deoxy- $\alpha$ -D-glucose, m.  $128-30^\circ$ , or the  $\text{D}$ -galactose analog, m.  $141-5^\circ$ , gave after acetylation the corresponding 6-acetamido-6-deoxy- $\alpha$ -D-glucosamine rather than septanose products with  $\text{N}$  in the ring. Redn. of 6-azido-6-deoxy- $\alpha$ -D-glucosamine, m.  $138-40^\circ$ , afforded 6-amino-6-deoxy- $\alpha$ -D-glucosamine, m.  $210-12^\circ$  (dec.  $\text{p.a.}$ ). Reaction of 2,3,4-O-isopropylidene-1,6-di-O- $\beta$ -D-xylo-1-fructofuranose with 1 molar equiv. of  $\text{NaN}_3$  in  $\text{HCON}_2$  at  $100^\circ$  for 18 hrs. gave 6-azido-6-deoxy-2,3-O-isopropylidene-1,6-di-O- $\beta$ -D-xylo-1-fructofuranose (I), m.  $70-82^\circ$ . Reductive rearrangement of the  $\beta$ -xylofuranosyl function in I by a benzylidene and subsequent decetylation and hydrolysis afforded 6-amino-6-deoxy- $\alpha$ -D-fructofuranose (II). Reductive rearrangement of I

gave 2-hydroxy-3,4,5-trihydroxypiperidine,  $[\alpha]_D^{25} -65^\circ$  (c 3  $\text{MeOH}$ ).  $\text{N}$ -acetyl deriv. m.  $198-9^\circ$ ,  $[\alpha]_D^{25} -11^\circ$  (c 1.55  $\text{MeOH}$ ).



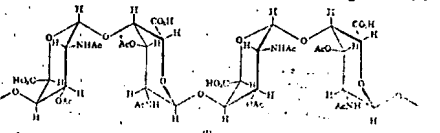
0.4  $\text{MeOH}$ ). Reductive rearrangement of 5-azido-5,6-dideoxy-1-talose and subsequent acetylation gave  $\text{N}$ -acetyl-6-methyl-3,4,5-trihydroxypiperidine,  $[\alpha]_D^{25} 11.4^\circ$  (c 1.55  $\text{MeOH}$ ).

**65782p** Sequence studies on *Diplococcus pneumoniae* type II polysaccharide. S. A. Barker, P. J. Somers, and M. Stacey (Univ. Birmingham, Eng.). *Carbohydr. Res.* 3 (3), 251-70 (1967) (Eng.). Assay of  $\text{HCO}_2\text{H}$  released by periodic oxidn. of *D. pneumoniae* Type II polysaccharide (SII); during liberation of an L-rhamnose-contg. trisaccharide from SII by  $\alpha$ -L-rhamnosidase, demonstrated an  $\alpha$ -(1  $\rightarrow$  4) linkage between this trisaccharide and the remainder of the SII mol. Carboxyl-reduced SII,  $[\alpha]_D^{25} 42^\circ$ , contg. 49.7%  $\beta$ -D-glucose and 49.8% L-rhamnose was prep'd. by redn. of the 2-hydroxyethyl ester in two stages. A comparison of the cleaved products of the action of specific, induced  $\alpha$ - and  $\beta$ -D-glucosidases on SII and carboxyl-reduced SII demonstrated the presence of O- $\beta$ -(D-glucopyranosyluronic acid)-(1  $\rightarrow$  4)-O- $\alpha$ -D-glucopyranosyl-(1  $\rightarrow$  4) residues in SII and O- $\beta$ -glucopyranosyl-(1  $\rightarrow$  4)-O- $\alpha$ -D-glucopyranosyl-(1  $\rightarrow$  4) residues in the reduced polysaccharide. A possible repeating unit for SII is discussed. RCYM

**65783q** Influence of the hydrogen ion concentration on the specificity of the TTC reaction. Klaus Lorenz and Hans Hoffmeister (Univ. Homburg, Ger.). *Mikrochim. Acta* 166 (6), 1062-7 (Ger.). Triphenyltetrazolium chloride (TTC), is widely used as a dehydrogenation reagent for org. compds. The  $\text{H}_2\text{O}:\text{sol. TTC}$  salt is converted during this reaction into the corresponding formazan, a red insol. compd. The spontaneous formation of formazan occurs only in alk. media and the number of compds. responding to dehydrogenation increases with increasing pH of the environment. The threshold pH of 12.45, above which TTC remains inactive, could be lowered to 12.0 by addn. of  $\text{MeOH}$  or *tert*- $\text{BuOH}$ . This shift reduces the no. of reagent partners, increases the specificity of the dehydrogenation, and allows one to follow quantit. the progress of reaction. Data on  $\beta$ -D-glucose,  $\beta$ -D-glucose 1-phosphate, sucrose, cellulose powder, sol. starch, glycogen, 2-amino-2-deoxy-D-glucose, hydrochloric acid, chondroitin sulfate, hyaluronic acid, heparin, Na lactate,  $\text{Na}_2\text{K}$  tartrate, Na pyruvate,  $\alpha$ -oxoglutaric acid, cysteine hydrochloride, creatinine, urea, and glycine are tabulated. G. W. Schief

**65784r** Preparation and characterization of 2-O-(4-O-methyl- $\alpha$ -D-glucopyranosyl)-D-xylopyranose. Nirmoleend Roy and T. B. Timell (State Univ., Coll. of Forestry, Syracuse, N.Y.). *Carbohydr. Res.* 3 (2), 240-7 (1966-1967) (Eng.). 2-O-(4-O-methyl- $\alpha$ -D-glucopyranosyluronic acid)-D-xylopyranose, prep'd. by partial acid hydrolysis of a (4-O-methylglucuronoxylan from aspen wood, was acetylated with  $\text{Ac}_2\text{O}:\text{NaOAc}$ . The product was reduced with  $\text{B}_2\text{H}_6$  in tetrahydrofuran and saponified with  $\text{NaOMe}$  to give 57% title compd., m.  $85-105^\circ$ ,  $[\alpha]_D^{25} 114^\circ$  ( $\text{H}_2\text{O}$ ). Derek Horton

**65785s** Elucidation of the structure of the Vi-antigen isolated from *Escherichia coli* 5390/38. K. Heyns and G. Kiesel (Univ. Hamburg, Ger.). *Carbohydr. Res.* 3 (3), 349-53 (1967) (Ger.). The structure of the Vi-antigen isolated from *E. coli* 5390/38 was elucidated by applying (1) methylation followed by redn., and (2) redn. prior to methylation. Hydrolyzates of the Vi-antigen material treated in these two ways contained 2-amino-deoxy-3-O-methyl-D-galactopyranose (from 1) and 2-amino-2-deoxy-3,6-di-O-methyl-D-galactopyranose (from 2), resp. The occurrence of these 2 compds., as well as the absence of 2-amino-2-deoxy-4-O-methyl-D-galactopyranose and 2-amino-2-deoxy-1,6-di-O-methyl-D-galactopyranose, conclusively demonstrates 1  $\rightarrow$  4 glycosidic bonds in the polysaccharide. The products formed by hydrolysis were quant. det'd. and identified using authentic



control substances in the amino acid analyzer of Moore, Spackman, and Stein by a special buffer. From these results a model of the structure of the Vi-antigen is presented. 20 references. RCYM